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SERIES I No. 18

# OFFICIAL GAZETTE



## GOVERNMENT OF GOA

### SUPPLEMENT

Department of Agriculture

#### ORDER

10-1-79/AGR Vol VIII

The following revised fertiliser (control) Order, 1985 issued by Government of India, Ministry of Agriculture and Rural Development (Department of Agriculture and Co-operation) New-Delhi which was published in extraordinary Gazette of India, Part - II Section 3 sub-section (1) is hereby republished in the Official Gazette for the General information of the public.

- (i) Fertiliser (Control) order 1985  
No. GSR 758 (E) dated 25/9/1985.

A. P. Panvelkar, Under Secretary (Agriculture)  
Panaji 2nd June, 1989.

#### MINISTRY OF AGRICULTURE AND RURAL DEVELOPMENT

(Department of Agriculture and Coopn.)  
New Delhi, the 25th September, 1985

#### ORDER

G.S.R. 758(E).- In exercise of the powers conferred by section 3 of the Essential Commodities Act, 1955 (10 of 1955), the Central Government hereby makes the following Order, namely:-

#### 1. Short title and commencement.

- (1) This Order may be called the Fer-

tiliser (Control) Order, 1985.

- (2) It shall come into force on the date of its publication in the Official Gazette.

#### 2. Definitions.-

In this Order, unless the context otherwise requires,-

- (a) "Act" means the Essential Commodities Act, 1955 (10 of 1955);
- (b) "certificate of source" means a certificate given by a State Government, Commodity Board, manufacturer, pool handling agency or, as the case may be, wholesale dealer indicating therein the source from which fertiliser for purpose of sale is obtained;
- (c) "Commodity Board" means the Coffee Board constituted under section 4 of the Coffee Act, 1942 (7 of 1942) or the Rubber Board constituted under section 4 of the Rubber Act, 1947 (24 of 1947) or the Tea-Board constituted under section 4 of the Tea Act, 1953 (29 of 1953), or, as the case may be, the Cardamom Board constituted under section 4 of the Cardamom Act, 1965 (42 of 1965);
- (d) "compound or complex fertiliser" means a fertiliser containing two or more essential plant nutrients during the production of which chemical reaction takes place;
- (e) "controller" means the person appointed as Controller of Fertilisers by the Central Government and includes any other person empowered by the Central

Government to exercise or perform all or any of the powers, or as the case may be, functions of the Controller under this Order;

- (f) "dealer" means a person carrying on the business of selling fertilisers, whether wholesale or retail, and includes a manufacturer and a pool handling agency carrying on such business and the agents of such person, manufacturer or pool handling agency;
- (g) "essential plant nutrients" include boron, calcium, carbon, chlorine, copper, hydrogen, iron, manganese, magnesium, molybdenum, nitrogen, oxygen, phosphorous, potassium, sulphur and zinc;
- (h) "fertiliser" means any substance intended to be used as a source of one or more essential plant nutrient and specified in column 2 of Part A of Schedule -I and includes a mixture of fertilisers and special mixture of fertilisers;
- (i) "Form" means a form appended to this Order;
- (j) "grade" means the nutrient element contents in the fertiliser expressed as a percentage of nitrogen (N), phosphoric acid ( $P_2O_5$ ) and/or potash ( $K_2O$ ) in that Order;
- (k) "granulated mixture" means a mixture of fertilisers containing any two or more essential plant nutrients made by intimately mixing two or more fertiliser materials with or without inert material and granulating them together, without involving any chemical reaction;
- (l) "inspector" means an Inspector of Fertilizers appointed under clause 27;
- (m) "manufacturer" means a person who produces fertilisers or mixtures of fertilisers and the expression 'manufacture' with its grammatical variations shall be construed accordingly;
- (n) "mixture of fertilisers" includes a physical mixture and a granulated mixture, but does not include organic fertilisers or re-enforced organic manures;
- (o) "offer for sale" includes a reference to an intimation by a person of

a proposal by him for the sale of any fertiliser, made by publication of a price list, by exposing the fertiliser for sale indicating the price, by furnishing of a quotation or otherwise howsoever;

- (p) "physical mixture" means a mixture of fertilisers containing any two or more essential plant nutrients made by physically mixing fertilisers with or without inert material necessary to make a required grade, without involving any chemical reaction;
- (q) "prescribed standard" means,-
  - (i) in relation to fertiliser included in column 1 of Part A of Schedule-I, the standard set out in the corresponding entry in column 2, subject to the limits of permissible variation as specified in Part B of that Schedule; and
  - (ii) in relation to a mixture of fertilisers, the standard set out in respect of that mixture under sub-clause (1) of clause 13 by the Central Government, subject to the limits of permissible variation as specified in Part B of Schedule-I;
- (r) "pool handling agency" means an agency entrusted by the Central Government with functions relating to handling and distribution of imported fertilisers;
- (s) "registering authority" means a registering authority appointed under clause 26;
- (t) "retail dealer" means a dealer who sells fertilisers to farmers/plantations;
- (u) "Schedule" means a Schedule appended to this Order;
- (v) "special mixture of fertilisers" means any mixture of fertilisers prepared for experimental purposes in pursuance of a requisition made by any person (including a person engaged in the cultivation of tea, coffee or rubber) for sale to that person in such quantity and within such period as may be specified in such requisition; and
- (w) "wholesale dealer" means a dealer who sells fertilisers otherwise than in retail.

## II. PRICE CONTROL

## 3. Fixation of prices of fertilisers.-

- (1) The Central Government may, with a view to regulating equitable distribution of fertilisers and making fertilisers available at fair prices, by notification in the Official Gazette, fix the maximum prices or rates at which any fertiliser may be sold by a dealer, manufacturer or a pool handling agency.
- (2) The Central Government may having regard to the local conditions of any area, the period of storage of fertilisers and other relevant circumstances, fixed different prices or rates for fertilisers having different periods of storage or for different areas or for different classes of consumers.
- (3) No dealer, manufacturer or pool handling agency shall sell or offer for sale any fertiliser at a price exceeding the maximum price or rate fixed under this clause.

## 4. Display of stock position and price list of fertilisers-

Every dealer, who makes or offers to make a retail sale of any fertilisers, shall prominently display in his place of business,-

- (a) the quantities of opening stock of different fertilisers held by him on each day;

Explanation-- The actual stocks at any point of time during the day may be different from that of the displayed opening stocks to the extent of sale and receipt of such fertilisers upto the time of inspection during that day.

- (b) a list of prices or rates of such fertilisers fixed under clause 3 and for the time being in force.

## 5. Issue of cash/credit memorandum.-

Every dealer shall issue a cash or credit memorandum to a purchaser of a fertiliser in such manner as the controller may direct.-

III. CONTROL ON DISTRIBUTION  
OF FERTILISERS BY  
MANUFACTURER

## 6. Allocation of fertilisers to various States.-

The Central Government may, with a view to securing equitable distribution and availability of fertilisers to the farmers in time, by notification in the Official Gazette, direct any manufacturer to sell the fertilisers produced by him in such quantities and in such State or States and within such period as may be specified in the said notification.

## IV. REGISTRATION OF DEALERS

## 7. Dealers to be registered.-

No person, including a manufacturer, a pool handling agency, a wholesale dealer and a retail dealer shall offer for sale or carry on the business of selling fertilisers at any place except under and in accordance with the terms and conditions of a certificate of registration granted to him under clause 9:

Provided that a State Government may, by notification in the Official Gazette, exempt from the provisions of this clause any person selling fertilisers to farmers in such areas and subject to such conditions as may be specified in that notification.

## 8. Application for registration.-

Every person desiring to obtain a certificate of registration under this Order for selling fertilisers, whether in wholesale or retail or both shall make an application for registration to the registering authority in Form A, in duplicate, together with the fee prescribed under clause 36 and a certificate of source:

Provided that where the applicant is a State Government, a manufacturer or a pool handling agency, it is not necessary to enclose a certificate of source along with the application:

Provided further that separate certificates of registration shall be obtained for selling fertiliser in whole-sale and retail:

Provided also that where fertilisers are obtained for sale from different sources, a certificate of source from each such source shall be furnished.

## 9. Grant of refusal of certificate of registration.-

The registering authority shall grant a certificate of registration in Form 'B' within thirty days of the receipt of application to any person who applies for it under clause 8:

Provided that no certificate of registration shall be granted to a person,-

- (a) if his previous certificate of registration is under suspension; or
- (b) if his previous certificate of registration has been cancelled within a period of one year immediately preceding the date of application;
- (c) if he has been convicted of an offence under the Act, or any Order made thereunder within three years immediately preceding the date of making the application; or
- (d) If he fails to enclose with the application a certificate of source; or
- (e) if the application is incomplete in any respect.

Period of validity of certificate of registration.-

Every certificate of registration granted under clause 9 shall, unless suspended or cancelled, be valid for a maximum period of three years, from the date of issue.

11. Renewal of certificate of registration.-

- (1) Every holder of a certificate of registration desiring to renew the certificate of registration granted under clause 9 shall, before the date of expiry of such certificate of registration make an application for renewal to the registering authority in Form C, in duplicate, together with the fees prescribed under clause 36 for such renewal and a certificate of source as required under clause 8.
- (2) On receipt of such application, together with such fee and certificate of source, the registering authority may renew the certificate of registration:

Provided that a certificate of registration shall not be renewed, if the holder of the certificate of registration did not sell any fertiliser during the period of one year immediately preceding the date of expiry of the period of validity of the certificate of registration sought to be renewed.

- (3) If any application for renewal is not made before the expiry of the period of validity of the certificate of registration but is made within one month from the date of such expiry, the certificate of registration may be renewed on payment of

such additional fee as may be prescribed by the State Government, in addition to the fee for renewal of the certificate of registration.

- (4) Where the application for renewal is made within the time specified in sub-clause (1) or sub-clause (3) the applicant shall be deemed to have held a valid certificate of registration, until such date as the registering authority passes orders on the application for renewal.
- (5) If an application for renewal of a certificate of registration is not made within one month of the date of expiry of the period of validity of the certificate of registration, the certificate of registration shall be deemed to have lapsed on the date on which its validity expired and any business carried on after that date shall be deemed to have been carried on in contravention of clause 7.

V. MANUFACTURE OF MIXTURES OF FERTILISERS

12. Restriction on preparation of mixtures of fertilisers.-

No person shall carry on the business of preparing any mixture of fertilisers or special mixture of fertilisers except under and in accordance with the terms and conditions of a certificate of manufacture granted to him under clause 16.

13. Standards of mixtures of fertiliser.-

- (1) Subject to the other provisions of this Order, no person shall manufacture any mixture of fertilisers unless such mixture conforms to the standards set out in the notification to be issued by the Central Government in the Official Gazette in so far as total nitrogen, neutral and ammoniumcitrate soluble phosphoric acid ( $P_2O_5$ ) water soluble phosphoric acid ( $P_2O_5$ ) and water soluble Potash ( $K_2O$ ) are concerned.
- (2) No certificate of manufacture shall be granted in respect of any mixture of fertilisers which does not conform to the standards set out in the notification referred to in sub-clause (1).
- (3) Nothing in this clause shall apply to special mixtures of fertilisers.

14. Application for certificate of manufacture of mixtures of fertilisers.-

- (1) Every person desiring to obtain a certificate of manufacture for preparation of any mixture of fertilisers or special mixture of fertilisers shall possess such qualification as may be prescribed for this purpose by the State Government or shall employ a person possessing such qualification for the preparation of such mixture.
- (2) An applicant for a certificate of manufacture for preparation of mixture of fertilisers or special mixture of fertilisers shall make an application to the registering authority.
  - (a) if he is an applicant for a certificate of manufacture for any mixture of fertilisers, in Form D, in duplicate, together with the fee prescribed therefor under clause 36; or,
  - (b) if he is an applicant for a certificate of manufacture for any special mixture, in Form E in duplicate together with the fee prescribed therefor under the said clause 36 and an attested copy of the requisition of the purchaser.

15. Grant or refusal of certificate of manufacture for preparation of mixtures of fertilisers.-

- (1) The registering authority may, for reasons to be recorded in writing, refuse to grant a certificate of manufacture in respect of any mixture of fertilisers of special mixture of fertilisers and shall furnish to the applicant a copy of the order so passed.
- (2) Where an application for a certificate of manufacture for mixture of fertilisers is not refused under sub-clause (1), the registering authority shall grant a certificate of manufacture in Form F and where an application for a certificate of manufacture for a special mixture is not refused under that sub-clause, such authority shall grant a certificate of manufacture to the applicant in Form G.

16. Conditions for grant of certificate of manufacture in respect of special mixture of fertilisers and period of validity of such certificate.-

- (1) No certificate of manufacture in respect of any special mixture of fertilisers shall be granted to an applicant unless he holds a valid certificate of manufacture under this Order for any mixture of fertilisers.
- (2) Every certificate of manufacture granted in respect of any special mixture of fertilisers shall be valid for a period of three months from the date of its issue:

Provided that the registering authority may, if it is satisfied that it is necessary so to do, extend the said period to such further period or periods as it may deem fit, so however, that the total period or periods so extended shall not exceed six months.

17. Period of validity of a certificate of manufacture for preparation of mixtures of fertilisers.-

Every certificate of manufacture granted under clause 15 for preparation of a mixture of fertilisers shall, unless suspended or cancelled, be valid for a period of three years from the date of issue.

18. Renewal of certificate of manufacture for preparation of mixtures of fertilisers.

- (1) Every holder of a certificate of manufacture for preparation of a mixture of fertilisers, desiring to renew the certificate, shall, before the date of expiry of the said certificate of manufacture make an application to the registering authority in Form D in duplicate, together with the fee prescribed for this purpose under clause 36.
- (2) On receipt of an application for renewal as provided in sub-clause (1), and keeping in view the performance of the applicant and other relevant circumstance, the registering authority may, if he so decides renew the certificate of registration by endorsement on Form F and in case the certificate of registration is not renewed, the registering authority shall record

in writing his reasons for not renewing the certificate of registration.

- (3) If an application for renewal is not made before the expiry of the certificate of registration but is made within one month from the date of expiry of the certificate of registration the certificate of registration may be renewed on payment of such additional fee as may be prescribed by the State Government for this purpose.
- (4) Where the application for renewal is made within the time specified in sub-clause (1) or sub-clause (3), the applicant shall be deemed to have held a valid certificate or registration until such date as the registering authority passes order on the application for renewal.
- (5) If an application for renewal of a certificate of manufacture is not made within the period stipulated under sub-clause (1) or, as the case may be, under sub-clause (3), the certificate of manufacture shall be deemed to have expired immediately on the expiry of its validity period, and any business carried on after that date shall be deemed to have been carried on in contravention of clause 12.

#### VI. RESTRICTIONS ON MANUFACTURE, SALE, ETC. OF FERTILISERS

##### 9. Restriction on manufacture, sale and distribution of fertilisers.-

- (1) No person shall himself or by any other person on his behalf-
- (a) manufacture for sale, sell, offer for sale, stock or exhibit for sale or distribute any fertiliser which is not of prescribed standard;
- (b) manufacture for sale, sell, offer for sale, stock or exhibit for sale, or distribute any mixture of fertilisers which is not of prescribed standard or special mixture of fertilisers which [subject to such limits of permissible variation in relation

to total nitrogen, neutral and ammonium citrate soluble phosphoric acid ( $P_2O_5$ ), water soluble phosphoric acid ( $P_2O_5$ ) or water soluble Potash ( $K_2O$ ) as may be specified from time to time by the Central Government does not conform to the particulars specified in the certificate of manufacture granted to him under this Order in respect of such special mixture.

- (c) sell, offer for sale, stock or exhibit for sale or distribute-
- (i) any fertiliser the container whereof is not packed and marked in the manner laid down in this Order;
- (ii) any fertiliser which is an imitation or a substitute for another fertiliser under the name of which it is sold;
- (iii) any fertiliser which is adulterated;

Explanation,- A fertiliser shall be deemed to be adulterated, if it contains any substance the addition of which is likely to eliminate or decrease its nutrient contents or make the fertiliser not conforming to the prescribed standard.

- (iv) any fertiliser the label or container whereof bears the name of any individual firm or company purporting to be manufacturer of the fertilizer, which individual, firm or company is fictitious or does not exist;
- (v) any fertilizer, the label or container whereof or anything accompanying therewith bears any statement which makes a false claim for the fertilizer or which is false or misleading in any material particular;
- (vi) any substance as a fertiliser which substance is not, in fact, a fertiliser; or
- (vii) any fertiliser without exhibiting the minimum guaranteed percentage by

weight of plant nutrient.

20. Specifications in respect of imported fertilisers.

Notwithstanding anything contained in this Order, the Central Government may, by an order published in the Official Gazette, fix separate specifications in respect of imported fertilisers.

21. Manufacturers pool handling agencies to comply with certain requirements in regard to packing and marking, etc.-

(1) Every manufacturer and pool handling agency shall in regard to packing and marking of containers of fertilisers, comply with the following requirements, namely:-

(a) Every container in which any fertiliser is packed shall bear such particulars as may from time to time be specified by the controller in this behalf, and

(b) every container shall be so packed and sealed that the contents thereof can not be tampered with without breaking the seal:

Provided that where fertiliser manufactured in India are packed in bags stitched in hand, such bags shall bear lead seals, so that the contents thereof cannot be tampered with without breaking the seals:

Provided further that lead sealing shall not be necessary,-

(i) if such bags are machine stitched in such a manner that contents thereof can not be tampered with without a visible break in the stitching; and

(ii) in the case of fertilisers imported from abroad and packed in bags stitched in hand, in such a manner that the contents thereof can not be tampered with without visible break in the stitching.

22. Bulk sale of fertilisers.-

Notwithstanding anything contained in this Order,-

(a) a retail dealer may retain at any time one bag or container of each variety of fertiliser in an open and unsealed condition for the purpose of sale;

(b) a manufacturer may sell the fertiliser manufactured by him in bulk to a manufacturer of mixture of fertilisers, compound/complex fertilisers or special mixture of fertilisers; and

(c) the Central Government may by notification published in the Official Gazette in this behalf authorise a manufacturer to sell any fertiliser manufactured by him in bulk also direct to farmers for such period as may be specified in that notification:

Provided that a certificate indicating the minimum guaranteed percentage of plant nutrients is issued by the manufacturer to each farmer at the time of such sale.

23. Disposal of non-standard fertilisers.-

(1) Notwithstanding anything contained in this Order, a person may sell, offer for sale, stock or exhibit for sale or distribute any fertiliser which, not being an adulterated fertiliser, does not conform to the prescribed standard (hereinafter in this Order referred to as 'non-standard fertiliser') subject to the conditions that,

(a) the container of such non-standard fertiliser is conspicuously superscribed in red colour with the words "non-standard" and also with the sign "X"; and

(b) an application for the disposal of non-standard fertilisers in Form H is submitted to the registering authority to grant a certificate of authorisation for sale of such fertilisers and a certificate of authorisation with regard to their disposal and price is obtained in Form I;

(c) such non-standard fertiliser shall be sold only to the manufacturers of mixtures of fertilisers or special mixtures of fertilisers or research forms of Government or universities or such bodies.

(2) The price per unit of the non-standard fertiliser shall be fixed by the registering authority after satisfying itself that the sample taken is a representative one, and after considering the nutrient contents in the sample determined on the basis of a chemical analysis of the non-standard fertiliser.

- (3) The Central Government may, by notification in the Official Gazette and subject to the conditions, if any, laid down in that notification, and subject to guidelines issued in this regard by the Central Government exempt such pool handling agencies, as it deems fit, from complying with conditions laid down in paragraphs (a) and (b) of the sub-clause (1).

24. Manufacturers/Pool handling agencies to appoint officers responsible with compliance of the order.

Every manufacturing organisation and pool handling agency shall appoint in that organisation and in consultation with the Central Government, an officer, who shall be responsible for compliance with the provisions of this Order.

25. Restriction on sale/use of fertilisers.

No person shall, except with the prior permission of the Central Government and subject to such terms and conditions as may be imposed by such Government; shall or use fertiliser, for purposes other than fertilisation of soils and increasing productivity of crops:

Provided that the price of fertilisers permitted for sale for industrial use shall be no profit no loss price, excluding all subsidies at the production, import, handling or on sale for agricultural consumers;

Provided further that wherever customs or excise duties are chargeable, these may be added to the price so fixed;

Provided also that in the case of non-standard fertilisers, reductions shall be made from the no profit no loss price, indicated above, proportionate to the loss of nutrient contents.

VII. ENFORCEMENT AUTHORITIES .-

26. Appointment of registering authority.-

The State Government may, by notification in the Official Gazette, appoint such number of persons, as it thinks necessary, to be registering authorities for the purpose of this Order, and may, in any such notification, define the limits of local area within which each such registering authority shall exercise his jurisdiction.

27. Appointment of inspectors.-

The State Government, or the Central Government may, by notification in the Official Gazette appoint such number of persons, as it thinks necessary, to be inspectors of fertilisers for the purpose of this Order, and may, in any such notification, define the limits of local area within which each such inspector shall exercise his jurisdiction.

28. Powers of inspectors.-

(1) An inspector may, may with a view to securing compliance with this Order,-

- (a) require any manufacturer, pool handling agency, wholesale dealer or retail dealer to give any information in his possession with respect to the manufacture, storage and disposal of any fertiliser manufactured or, in any manner handled by him;
- (b) draw samples of any fertiliser in accordance with the procedure of drawing of samples laid down in Schedule II;

Provided that the inspector shall prepare the sampling details in duplicate in Form J, and hand over one copy of the same to the dealer or his representative from whom the sample has been drawn:

- (c) enter upon and search any premises where any fertiliser is manufactured or stored or exhibited for sale, if he has reason to believe that any fertiliser has been or is being manufactured, sold, offered for sale, stored, exhibited for sale or distributed contrary to the provisions of this Order;
- (d) seize or detain any fertiliser in respect of which he has reason to believe that a contravention of this Order has been or is being or is about to be committed;
- (e) seize any books of accounts of documents relating to manufacture, storage or sale of fertilisers, etc. in respect of which he has reason to believe that any contravention of this Order has been or is being or is about to be committed;

Provided that the inspector shall give a receipt for such fertilisers or books of accounts or documents so seized



to the person from whom the same have been seized:

Provided further that the books of accounts or documents so seized shall be returned to the person from whom they were seized after copies thereof or extracts therefrom, as certified by such person, have been taken.

(2) Subject to the proviso to paragraphs (d) and (e) of sub-clause (1), the provisions of the Code of Criminal Procedure, 1973 (2 of 1974) relating to search and seizure shall, so far as may be, apply to searches and seizures under this clause.

(3) Where any fertiliser is seized by an inspector under this clause, he shall forthwith report the fact of such seizure to the collector whereupon the provisions of sections 6A, 6B, 6C, 6D and 6E of the Act, shall apply to the custody, disposal and confiscation of such fertilisers.

(4) Every person, if so required by an inspector, shall be bound to afford all necessary facilities to him for the purpose of enabling him to exercise his powers under sub-clause (1).

#### VIII ANALYSIS OF SAMPLES

##### 29. Laboratory for analysis.-

A fertiliser sample, drawn by an inspector, shall be analysed in accordance with the instructions contained in Schedule II in the Central Fertiliser Quality Control and Training Institute or in any other laboratory notified for this purpose by the State Government.

##### 30. Time limit for analysis, and communication of result.-

- (1) Where sample of a fertiliser has been drawn, the same shall be despatched, along with a memorandum in Form K to the laboratory for analysis within a period of seven days from the date of its drawal.
- (2) The laboratory shall analyse the sample and forward the analysis report in Form L within 60 days from the date of receipt of the sample in the laboratory to the authority specified in the said memorandum.
- (3) The authority to whom the analysis report is sent under sub-clause (2)

shall communicate the result of the analysis to the dealer/manufacturer/pool handling agency from whom the sample was drawn within 30 days from the date of receipt of the analysis report of the laboratory.

#### IX. MISCELLANEOUS

##### 31. Suspension/cancellation of registration certificate.-

- (1) A registering authority may, after giving the holder of a certificate of registration or a certificate of manufacture or any other certificate granted under this Order, an opportunity of being heard, suspend or cancel such certificate on any of the following grounds, namely:-

- (a) that such certificate has been obtained by mis-representation as to material particulars;
- (b) that any of the provisions of this Order or any of the terms and conditions of such certificate has been contravened or not fulfilled:

Provided that while cancelling the certificate the holder thereof may be allowed a period of 30 days to dispose of the balance stock of fertilisers if any, held by him:

Provided further that the stock of fertiliser lying with the holder after the expiry of the said 30 days shall be confiscated.

- (2) Where the contravention alleged to have been committed by a person is such as would, on being proved, justify cancellation of the certificate of registration or, as the case may be, certificate of manufacture or any other certificate granted under this Order to such person the registering authority may, without any notice, suspend such certificate, as an interim measure:

Provided that the registering authority shall immediately furnish to the person details and the nature of contravention alleged to have been committed by such person and, after giving the person an opportunity of being heard, pass final orders either revoking the order of suspension or cancelling the certificate within fifteen days from the date of

issue of the order of suspension:

Provided further that where no final order is passed within the period as specified above, the order of interim suspension shall be deemed to have been revoked without prejudice, however, to further action which the registering authority may take against the holder of the certificate under sub-clause (1).

(3) Wherever a certificate is suspended or cancelled under this clause, the registering authority shall record a brief statement of the reasons for such suspension or, as the case may be, cancellation and furnish a copy thereof to the person whose certificate has been suspended or cancelled.

32. Appeal.-

Any person aggrieved by-

(1) an order

- (a) refusing to grant, amend or renew a certificate of registration for sale of fertilisers; or
- (b) refusing to grant a certificate of manufacture for preparation of mixture of fertilisers or special mixture of fertilisers; or
- (c) suspending or cancelling a certificate of registration or manufacture; or

(2) non-issuance of certificate of registration to him within the stipulated period may within sixty days from the date of receipt of such order or, as the case may be, from the date of expiry of such stipulated period, appeal to such authority as the State Government may specify in this behalf, and the decision of such authority shall be final.

33. Grant of duplicate copies of certificate of registrations etc.-

Where a certificate of registration or a certificate of manufacture or any other certificate granted or, as the case may be, renewed under this Order is lost, the registering authority may, on an application made in this behalf, together with the fee prescribed for this purpose under clause 36, grant a duplicate copy of such certificate.

34. Amendment of certificate of registration.-

The registering authority may, on an application made in this behalf, together with the fee prescribed for the purpose under clause 36, amend a certificate of registration.

35. Maintenance of records and submission of returns, etc.-

(1) The Controller may by an order made in writing direct the dealers, manufacturers, and pool handling agencies-

- (a) to maintain such books of accounts, records etc. relating to their business and in such form, as may be specified in that order; and
- (b) to submit to such authority, returns and statements in such form and containing such information relating to their business and within such time as may be specified in that order.

(2) Where person holds certificates of registration for retail sale and wholesale sale of fertilisers, he shall maintain separate books of accounts for these two types of sales made by him.

36. Fees.-

(1) The fees payable for grant, amendment or renewal of a certificate of registration or certificate of manufacture a duplicate of such certificates or renewal thereof under this Order shall be such as the State Government may, with prior approval of the Controller, from time to time fix, subject to the maximum fees fixed for different purposes by the Central Government and different fees may be fixed for different purposes or for different classes of dealers or for different types of mixtures of fertiliser or special mixture:-

Provided that no fee shall be payable by a person holding a valid certificate of manufacture for preparation of mixture of fertilisers or special mixture of fertiliser issued under the Fertiliser (Control) Order, 1957 and making an application for grant of a fresh certificate of manufacture for preparation of such mixture after the commencement of this Order.

(2) The authority to whom and the manner in which the fee fixed under sub-clause (1) shall be paid shall be such as may be specified by the State Government by notification in the Official Gazette.

(3) Any fee paid under sub-clause (1) shall not be refundable unless the grant or renewal of any certificate of registration or certificate of manufacture or duplicate copy of such certificate or renewal under this Order has been refused.

### 37. Service of orders and directions.-

Any order or direction made or issued by the controller or by any other authority under this order shall be served in the same manner as provided in sub-section (5) of section 3 of the Act.

### 38. Advisory Committee.-

(1) The Central Government may by notification in the Official Gazette and on such terms and conditions as may be specified in such notification, constitute a Committee called the Central Fertiliser Committee consisting of a Chairman and not more than ten other persons, having experience or knowledge in the field, who shall be members of the Committee, to advise the Central Government regarding-

- (i) inclusion of a new fertiliser, under this Order;
- (ii) specifications of various fertilizers;
- (iii) grades/formulations of physical granulated mixtures of fertilisers that can be allowed to be prepared in a state;
- (iv) requirements of laboratory facilities in a manufacturing unit, including a unit manufacturing physical/granulated mixtures of fertilisers;
- (v) methods of drawal and analysis of a sample;
- (vi) any other matter referred by the Central Government to the Committee.

(2) The Committee may, subject to the previous approval of the Central Government, make by-laws fixing the quorum and regulation its own procedure and the conduct of all business to be transacted by it.

(3) The Committee may co-opt such number of experts and for such purposes or periods as it may deem fit, but any expert so co-opted shall not have the right to vote.

(4) The Committee may appoint one or more sub-committees, consisting wholly of members of the Committee or wholly of co-opted members or partly of the members of the Committee and partly of co-opted members, as it thinks fit, for the purpose of discharging such of

its functions as may be delegated to such sub-committee or sub-committees by the Central Fertiliser Committee.

### 39. Repeal and saving.-

(1) The Fertiliser (Control) Order, 1957 is hereby repealed except as respects things done or omitted to be done under the said Order before the commencement of this Order.

(2) Notwithstanding such repeal, an order made by any authority, which is in force immediately before the commencement of this Order and which is consistent with this Order, shall continue in force and all appointments made, prices fixed, certificates granted and directions issued under repealed Order and in force immediately before such commencement shall likewise continue in force and be deemed to be made fixed, granted or issued in pursuance of this order till revoked.

## SCHEDULE I

[See clauses 2(h)&(q)]

### Part - A. Specifications of fertilisers

Name of the Fertiliser	Specifications	
1	2	
1. (a) Straight Nitrogenous Fertilisers:		
1. Ammonium Sulphate	(i) Moisture per cent by weight, maximum	1.0
	(ii) Ammoniacal nitrogen per cent by weight, minimum	20.6
	(iii) Free acidity (as $H_2SO_4$ ) per cent by weight maximum (0.04 for material obtained from by product ammonia and by-product gypsum)	0.025
	(iv) Arsenic ( $As_2O_3$ ) per cent by weight, maximum	0.01
2. Urea (46%N) While free flowing)	(i) Moisture per cent weight, maximum	
	(ii) Total nitrogen, per cent by weight, (on dry basis) minimum	45.00
	(iii) Biuret per cent by weight, maximum	1.5

1	2		
		(iv) Particle size-90 per cent of the material shall pass through 2.8 mm IS sieve and not less than 80 per cent by weight shall be retained on 1 mm IS sieve.	retained on 1 mm IS sieve. Not more than 10 per cent shall be below 1 mm IS sieve.
3. Urea (coated) (45%N) (While free flowing)		(i) Moisture per cent by weight, maximum 0.5	6. Calcium Ammonium Nitrate (25%N) (i) Moisture per cent by weight, maximum 1.0
		(ii) Total nitrogen per cent by weight content with coating minimum 45.0	(ii) Total ammoniacal and nitrate nitrogen per cent by weight, minimum 26.0
		(iii) Biuret per cent by weight maximum 1.5	(iii) Ammoniacal nitrogen per cent by weight, minimum 13.0
		(iv) Particle size-90 per cent of the material shall pass through 2.8 mm IS sieve and not less than 80 per cent by weight shall be retained on 1 mm IS sieve	(iv) Calcium nitrate per cent by weight, maximum 0.5
4. Ammonium Chloride		(i) Moisture per cent by weight, maximum 2.0	(v) Particle size - 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve
		(ii) Ammoniacal nitrogen per cent by weight, minimum 25.0	7. Anhydrous Ammonia (i) Ammonia per cent by weight, minimum 99.0
		(iii) Chloride other than ammonium chloride (as NaCl) per cent by weight (on dry basis) maximum	(ii) Water per cent by weight, maximum 1.0
		(iv) Particle size 90 per cent material shall pass through 2.8 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.	(iii) Oil content by weight, maximum 20 ppm
5. Calcium Ammonium Nitrate (25%N)		(i) Moisture per cent by weight, maximum 1.00	1. (b) Straight Phosphatic fertilisers:
		(ii) Total ammoniacal and nitrate nitrogen per cent by weight, minimum 25.0	1. Single Super-phosphate (16% P <sub>2</sub> O <sub>5</sub> Powdered) (i) Moisture, per cent by weight, maximum 12.0
		(iii) Ammoniacal nitrogen per cent by weight minimum 12.5	(ii) Free phosphoric acid (as P <sub>2</sub> O <sub>5</sub> ) Per cent by weight, maximum 4.0
		(v) Calcium nitrate per cent by weight, maximum 0.5	(iii) Water soluble phosphates (as P <sub>2</sub> O <sub>5</sub> ) per cent by weight, minimum 16.0
		(v) Particle size-80 per cent of the material shall pass through 4 mm IS sieve and be	2. Single Superphosphate (14% P <sub>2</sub> O <sub>5</sub> powdered) (i) Moisture, per cent by weight, maximum 12.0
			(ii) Free phosphoric acid (as P <sub>2</sub> O <sub>5</sub> ), per cent by weight, maximum 4.0
			(iii) Water soluble phosphates (as P <sub>2</sub> O <sub>5</sub> ) per cent by weight minimum 14.0
			3. Triple Superphosphate (i) Moisture, per cent by weight, maximum 12.0
			(ii) Free phosphoric acid (as P <sub>2</sub> O <sub>5</sub> ) per cent by weight, maximum 3.0
			(iii) Total phosphates (as P <sub>2</sub> O <sub>5</sub> ) per cent by weight, minimum 46.0
			(iv) Water soluble phosphates 42.5

(1)		(2)		(1)		(2)	
	(as $P_2O_5$ ) per cent by weight, minimum				cent by weight (on dry basis) maximum.		
4. Bone meal, Raw	(i) Moisture per cent by weight, maximum	8.0		(iv) Particle size-95 per cent of the material shall pass through 1.7 mm IS sieve and be retained on 0.25 mm IS sieve.			
	(ii) Acid insoluble matter, per cent by weight, maximum	12.0					
	(iii) Total phosphates (as $P_2O_5$ ) per cent by weight, minimum	20.0	2. Potassium Sulphate	(i) Moisture per cent by weight, maximum.	1.5		
	(iv) 2 per cent citric acid soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum	8.0		(ii) Potash content (as $K_2O$ ) per cent by weight, minimum.	48.0		
	(v) Nitrogen content of water insoluble portion, per cent by weight, minimum.	3.0		(iii) Total chlorides (as Cl) per cent by weight (on dry basis) maximum.	2.5		
	(vi) Particle size: The material shall pass wholly through 2.36 mm IS sieve of which not more than 30 per cent shall be retained on 0.85 mm IS sieve.			(iv) Sodium as NaCl per cent by weight (on dry basis) maximum.	2.0		
5. Bone meal, Steamed	(i) Moisture, per cent by weight, maximum.	7.0	3. Potassium Schoenite	(i) Moisture per cent by weight, maximum	1.5		
	(ii) Total phosphates (as $P_2O_5$ ) per cent by weight (on dry basis) minimum.	22.0		(ii) Potash content (as $K_2O$ ) per cent by weight (on dry basis) minimum.	23.0		
	(iii) 2 per cent citric acid soluble phosphates (as $P_2O_5$ ) per cent by weight (on dry basis) minimum.	16.0		(iii) Magnesium oxide (as MgO) per cent by weight, maximum	10.0		
	(iv) Particle size-Not less than 90 per cent of the material shall pass through 1.18 mm IS sieve.			(iv) Total chlorides (as Cl) (on dry basis) per cent by weight, maximum.	2.5		
6. Rock-phosphate	(i) Particle size-minimum 90 per cent of the material shall pass through 0.15 mm IS sieve and the balance 10 per cent of material shall pass through 0.25 mm IS sieve.			(v) Sodium (as NaCl) (on dry basis) per cent by weight, maximum	1.5		
	(ii) Total $P_2O_5$ content to be guaranteed by dealers.		1. (d) N.P Fertilisers				
1. (c) Straight potassic fertilisers	(i) Moisture per cent by weight, maximum.	0.5	1. Diammonium Phosphate (18-46-0)	(i) Moisture, per cent by weight, maximum.	1.0		
	(ii) Water soluble potash content (as $K_2O$ ) per cent by weight, minimum	58.0		(ii) Total nitrogen all in ammoniacal form per cent by weight, minimum	18.0		
1. Potassium Chloride (Muriate of potash)	(iii) Sodium as NaCl per-	3.5		(iii) Total phosphates (as $P_2O_5$ ) per cent by weight, minimum	46.0		
				(iv) Water soluble phosphates (as $P_2O_5$ ), per cent by weight, minimum.	41.0		
			2. Diammonium Phosphate with urea (18-46-0)	(i) Moisture per cent by weight, maximum.	1.5		
				(ii) Total nitrogen per cent by weight, minimum.	18.00		
				(iii) Ammoniacal nitrogen form per cent by weight, minimum	15.5		
				(iv) Total nitrogen in the form of urea per cent by weight, maximum.	2.5		

(1)		(2)		(1)		(2)	
	(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum	46.0		(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum		17.0	
	(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	41.0		(vii) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and shall be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			
	(vii) Particle size-90 per cent of the material shall pass through 4mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below than 1 mm size.						
3. Ammonium phosphate Sulphate (16-20-0)			5. Ammonium phosphate Sulphate nitrate (20-20-0)	(i) Moisture per cent by weight, maximum		1.5	
	(i) Moisture per cent by weight, maximum.	1.0		(ii) Total nitrogen per cent by weight, minimum		20.0	
	(ii) Total ammoniacal nitrogen per cent by weight, minimum.	16.0		(iii) Ammoniacal nitrogen percent by weight, minimum		17.0	
	(iii) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent weight, minimum.	20.0		(iv) Nitrate nitrogen per cent by weight, maximum		3.0	
	(iv) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	19.5		(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum		20.0	
	(v) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and shall be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum		17.0	
4. Ammonium phosphate Sulphate (20-20-0)				(vii) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and shall be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			
	(i) Moisture per cent by weight maximum,	1.0	6. Ammonium, phosphate Sulphate (18-9-0)	(i) Moisture per cent by weight maximum		1.0	
	(ii) Total nitrogen per cent by weight minimum	20.0		(ii) Ammoniacal nitrogen per cent by weight, minimum		18.0	
	(iii) Ammoniacal nitrogen per cent by weight minimum	18.0		(iii) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum		9.0	
	(iv) Nitrogen in the form of urea per cent by weight, maximum.	2.0		(iv) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum		8.5	
	(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	20.0		(v) Particle size 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			
			7. Nitro Phosphate	(i) Moisture per cent by weight, maximum.		1.5	

(1)		(2)		(1)		(2)	
(20-20-0)		(ii) Total nitrogen per cent by weight, minimum	20.0			of urea per cent by weight, maximum.	
		(iii) Nitrogen in ammoniacal form per cent by weight, minimum.	10.0			(v) Neutral ammonium citrate soluble phosphates (as P O ) per cent by weight, minimum.	24.0
		(iv) Nitrogen in nitrate form per cent by weight, maximum	10.0			(vi) Water soluble phosphate (as P O ) per cent by weight, minimum.	20.4
		(v) Neutral ammonium citrate soluble phosphates (as P O ) per cent by weight, minimum.	20.0			(vii) Particle size- per cent of the material shall pass through 4 mm IS sieve Not more than 5 per cent shall be below 1 mm IS sieve.	
		(vi) Water soluble phosphates (as P O ) per cent by weight, minimum	5.4			(Note: The products contains inert filler material such as sand or dolomite to the extent of 20% by weight maximum),	
		(vii) Calcium Nitrate per cent by weight, maximum	1.0				
		(viii) Particle size: 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.		10. Urea Ammonium Phosphates (20-20-0)	(i) Moisture per cent by weight, maximum.	1.5	
8. Urea ammonium phosphate (28-28-0)	(i) Moisture per cent by weight, maximum.	1.5			(ii) Total nitrogen per cent by weight, minimum.	20.0	
	(ii) Total nitrogen per cent by weight, minimum	28.0			(iii) Ammoniacal nitrogen per cent by weight, minimum	6.4	
	(iii) Ammoniacal nitrogen per cent by weight, minimum	9.0			(iv) Neutral ammonium citrate soluble phosphates (as P O ) per cent by weight, minimum.	20.0	
	(iv) Neutral ammonium citrate soluble phosphate (as P O ) per cent by weight, minimum.	28.0			(v) Water soluble phosphates (as P O ) per cent by weight, minimum.	17.0	
	(v) Water soluble phosphates (as P O ) per cent by weight, minimum	25.2			(vi) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not mote than 5 per cent shall be below 1 mm IS sieve.		
	(vi) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.				(Note: This product contains filler material (inert soil) to the extent of 30 per cent by weight/		
9. Urea Ammonium Phosphate (24-24-0)	(i) Moisture per cent by weight, maximum.	1.5		11. Mono ammonium phosphate (11-52-0)	(i) Moisture per cent by weight, maximum.	1.0	
	(ii) Total nitrogen per cent by weight, minimum	24.0			(ii) Total nitrogen all in ammoniacal form per cent by weight, minimum.	11.0	
	(iii) Ammoniacal nitrogen, per cent by weight, minimum.	7.5			(iii) Neutral ammonium citrate soluble phosphates (as P O ) per cent by weight, minimum.	52.0	
	(iv) Nitrogen in the form.	16.5			(iv) Water soluble phosphates (as P O ) per cent by weight minimum	44.2	
					(v) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS		

(1)	(2)	(1)	(2)
	sieve. Not more than 5 per cent shall be below 1 mm IS sieve.		$P_2O_5$ per cent by weight, minimum.
(e) N.P.K. Fertiliser:		(iv)	Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 27.2
Nitrophosphate with Potash (15-15-15)	(i) Moisture, per cent by weight, maximum 1.5 (ii) Total nitrogen minimum. 15.0 (iii) Ammoniacal nitrogen, per cent by weight, minimum 7.5 (iv) Nitrate nitrogen, per cent by weight, maximum. 7.5 (v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight minimum. 15.0 (vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight minimum. 4.0 (vii) Water soluble Potash (as $K_2O$ ) per cent by weight minimum. 15.0 (viii) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. (ix) Calcium nitrate, per cent by weight, maximum 1.0	(v)	Water soluble potash (as $K_2O$ ) per cent by weight, minimum. 16.0 (vi) Particle size-90 per cent material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.
N.P.K. (10-26-26)	(i) Moisture, per cent by weight, maximum. 1.0 (ii) Nitrogen in ammoniacal form, per cent by weight, minimum. 10.0 (iii) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 26.0 (iv) Water soluble potash (as $K_2O$ ) per cent by weight, minimum. 26.0 (v) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 22.1 (vi) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.	4. N.P.K. (22-22-11)	(i) Moisture per cent by weight, maximum. 1.5 (ii) Total nitrogen, per cent by weight, minimum. 22.0 (iii) Ammoniacal nitrogen per cent by weight, minimum. 7.0 (iv) Urea nitrogen, per cent by weight, maximum. 15.0 (v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 22.0 (vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 18.7 (vii) Water soluble potash (as $K_2O$ ) per cent by weight, minimum. 11.0 (viii) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.
N.P.K. (12-32-16)	(i) Moisture, per cent by weight, maximum. 1.0 (ii) Nitrogen in ammoniacal form, per cent by weight minimum. 12.0 (iii) Neutral ammonium citrate soluble phosphates (as	5. N.P.K. (14-35-14)	(i) Moisture, per cent by weight, maximum. 1.0 (ii) Nitrogen in ammoniacal form per cent by weight, minimum. 12.0 (iii) Urea nitrogen, minimum. 2.0 (iv) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum. 35.0 (v) Water soluble phosphate (as $P_2O_5$ ) per cent by weight, minimum. 29.0



6. N.P.K. (17-17-17)	(vi) Water soluble potash (as $K_2O$ ) per cent by weight, minimum.	14.0	8. N.P.K. (19-19-19)	cent shall be below 1 mm IS sieve.	
	(vii) Particle size-90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			(i) Moisture, per cent by weight, maximum.	1.5
	(i) Moisture, per cent by weight, maximum	1.5		(ii) Total nitrogen, per cent by weight, minimum.	19.0
	(ii) Total nitrogen, per cent by weight, minimum	17.0		(iii) Ammoniacal nitrogen, per cent by weight, minimum	5.6
	(iii) Ammoniacal nitrogen, per cent by weight, minimum	5.0		(iv) Urea nitrogen, per cent by weight, maximum.	13.4
	(iv) Urea nitrogen, per cent by weight, maximum.	12.0		(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	19.0
	(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	17.0		(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	16.2
	(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	14.5		(vii) Water soluble potash (as $K_2O$ ) per cent by weight, minimum.	19.0
7. N.P.K. (14-28-14)	(vii) Water soluble potash (as $K_2O$ ) per cent by weight, minimum.	17.0	1. (f) Micro-nutrients	(viii) Particle size: 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.	
	(viii) Particle size: 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.			1. Zinc Sulphate	
	(i) Moisture, per cent by weight, maximum.	1.5		(i) Free flowing crystalline form.	
	(ii) Total nitrogen, per cent by weight, minimum.	14.0		(ii) Matter insoluble in water, per cent by weight, maximum.	1.0
	(iii) Ammoniacal nitrogen, per cent by weight, minimum.	8.0		(iii) Zinc (as Zn), per cent by weight, minimum.	21.0
	(iv) Urea nitrogen, per cent by weight, maximum.	6.0		(iv) Lead (as Pb), per cent by weight, maximum.	0.003
	(v) Neutral ammonium citrate soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	28.0		(v) Copper (as Cu), per cent by weight, maximum.	0.1
	(vi) Water soluble phosphates (as $P_2O_5$ ) per cent by weight, minimum.	23.8		(vi) Magnesium (as Mg), per cent by weight, maximum.	0.5
	(vii) Water soluble potash (as $K_2O$ ) per cent by weight, minimum.	14.0	2. Manganese Sulphate	(vii) pH	Not less than 4.0
	(viii) Particle size: 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per			(i) Free flowing form	
				(ii) Matter insoluble in water, per cent by weight, maximum.	1.2
				(iii) Manganese (as Mn) content per cent by weight, minimum.	30.5
			(iv) Lead (as Pb) per cent by weight, maximum.		
			(v) Copper (as Cu) per cent		

By Weight, maximum.

(vi) Magnesium (as Mg) per cent 2.0  
by weight, maximum.

(vii) pH 3.75 ± 0.25

Part - B. Tolerance limit in plant nutrient for various fertilisers.

1. For fertilisers with definite compounds like ammonium sulphate, urea, ammonium chloride, muriate of potash, sulphate or potash, super-phosphate, decalcium phosphate, which contain more than 20 per cent plant nutrients.	0.2
For those which contain less than 20 per cent plant nutrients.	0.1
2. For calcium ammonium nitrate	0.3
3. For diammonium phosphate, nitrophosphates ammonium sulphate nitrate, urea ammonium phosphate, ammonium phosphate sulphate, bone meal, granulated mixed fertilisers, compound fertilisers.	0.5 for indivi- dual nut- rients subject to the maximum of 2.5 per cent for all combined nutrients

## SCHEDULE - II

[See clause 28(1)(b) and 29]

### Part-A. PROCEDURE FOR DRAWAL OF SAMPLES OF FERTILISERS

#### 1. General requirements of sampling-

In drawing samples, the following measures and precautions should be observed:-

- Samples shall not be taken at a place exposed to rain/sun;
- The sampling instruments shall be clean and dry when used;
- The material being sampled, the sampling instrument and the bags of samples should be free from any adventitious contaminations.
- To draw a representative sample, the contents of each bag selected for sampling should be mixed as thoroughly as possible by suitable means;
- The sample should be kept in suitable, clean dry and air tight

glass or screwed hard Polythene bottle of about 400 gm capacity or in a thick guaged Polythene bag. This should be put in a cloth bag which may be sealed with the Inspector's seal after putting in side the detailed description as specified in form 'J'. Indentifiable details may also be put on the cloth bag Like sample No./code No. or any other details which enables its identification;

- Each sample bag should be sealed air tight after filling and marked with details of sample, type and brand of fertiliser, name of dealer/manufacturer and the name of Inspector who has collected sample.

#### 2. Sampling from bagged material

##### (i) Scale of sampling

- Lot (for manufacturers)-All bags in a single consignment of the material of the same grade and type drawn from a single batch of the manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, all the bags of each batch shall constitute a separate lot.

In the case of a consignment drawn from a continuous Process, 2000 bags (or 100 tonnes) of the material shall constitute a lot.

- Lot (for dealers)-The lot is an identifiable quantity of same grade and type of fertiliser stored at an identifiable place subject to a maximum limit of 100 tonnes.

The lot shall be identified by the inspector based on visible appearance of bags, their packing and storage conditions. The stock of less than 100 tonnes with a dealer may also constitute one or more lots, if the material (fertilizer) of different sources and brand is available in such quantities.

- Selection of bags for sampling-  
The number of bags to

be chosen from a lot shall depend upon the size of the lot as given in the table below:-

Lot size (N)	(No. of bags)	No. of bags to be selected for sampling (n)
Less than	10	1
	10-100	2
	100-200	3
	200-400	4
	400-600	5
	600-800	6
	800-1000	7
	1000-1300	8
	1300-1600	9
	1600-2000	10

All the bags of a lot should be arranged in a systematic manner. Start counting from any bag randomly, go on counting as 1,2,3, ... upto r and so on, r being equal to the integral of  $N/n$ . Thus every rth bag counted shall be withdrawn and all bags shall constitute the sample bags from where the sample is to be drawn for preparing a composite sample.

(ii) Sampling from big godown/high Stackings - If the Procedure given in para 2(i) (c) is not possible to be adopted the sample should be drawn from the randomly selected fertilizer bags from different layers, front top and from all open sides in a zig zag fashion.

(iii) Sampling from small godowns- All the fertilizer bags of the same grade and type of each manufacturer though received on different dates shall be segregated and properly stacked. All bags of same grade and type of fertilizer manufactured by a particular manufacturing unit may be considered as one lot based on their physical conditions and the sample shall be drawn as per procedure laid down in para 2(i)(c) and 4.

(iv) Sampling from damaged stock-

(a) in case of torn or lumpy bags, damaged fertilizer bags or sweepings, the stock should be arranged according to identifiable lots. From each lot the number of bags shall be selected as per procedure (2) (i) (c) if the bags allow the use of sampling probe conveniently, the samples should be drawn by sampling probe.

(b) in case it is not possible to use the sampling probe, the bags may be opened and fertilizer material mixed together

uniformly by hammering the big lumps or putting Pressure if required, and then samples drawn by using suitable sample device.

### 3. Sampling Probe-

(i) An appropriate sampling instrument to be used by the Inspectors for collection of a representative sample is called sampling probe. The probe may comprise of a slotted single tube with solid conetip made of stainless steel or brass. The length of the probe may be approximately 60 to 65 cms and the diameter of the tube may be approximately 1.5 cm and the slot width may be 1.2 to 1.3 cms. The probe may be used if the physical conditions of the fertilisers and the packing material permits its use.

(ii) In case of High Density Polyethylene Packings and also when the fertilizer material is not in free flowing condition, the use of sampling probe may not be possible. In such a case, selected bags for drawing samples may be opened and the fertilisers may be taken out of the bags and spread on a clean surface and a sample drawn with the help of a suitable sampling device which may be made of stainless steel or brass cup.

### 4. Drawal of samples from bags-

(i) Drawal of sample and preparation of composite samples. Draw, with an appropriate sampling instrument, (sampling probe) small portions of the material from the selected bags as per procedure in Para 2(i) (b), 2(ii), 2(iii) and 2(iv) (a). The sampling probe shall be inserted in the bag from one corner to another diagonally and when filled with fertilizer, the probe is withdrawn and fertilizer is emptied in a container/or on Polythene sheet/or on a clean hard surface and made into one composite sample.

(ii) If the bags do not permit the use of sampling probe empty the contents of the bags on a level, clean and hard surface and drawn a composite sample

by the process of quartering as described under para 3(ii) or 5.

5. Preparation of composite sample-

If the composite sample collected from the different selected bags is larger than about 1.5 kg in weight, its size shall be reduced by method of quartering as detailed below:-

Spread the composite sample on a level clean hard surface flatten it out and divide it into four equal parts. Remove any diagonally opposite parts. Mix the two remaining parts together to form a cone, flatten out the cone and repeat the operation of quartering till a composite sample of about 1.5 kg weight is obtained.

6. Preparation of test sample and reference sample:-

- (i) The composite sample obtained above shall be spread out on a clean, hard surface and divided into three approximately equal portions of about 400 gm each in weight. Each of these samples shall constitute the test sample.
- (ii) Each test sample shall be immediately transferred to a suitable container as defined under para 1(e). The slip with detailed description may be put inside the sample bag. Each bag shall also be properly labelled as mentioned in para 1(f).
- (iii) Each test sample container shall then be sealed with the seals of the inspector. If possible, seal of the manufacturer/dealer or purchaser as the case may be, may also be affixed.
- (iv) One sample so sealed shall be sent to the Incharge of the Laboratory notified by the State Government under clause 29 or Central Fertiliser Quality Control and Training Institute, Faridabad for analysis and the second given to the manufacturer or dealer or the purchaser as the case may be. The third sample shall constitute the reference sample and shall be sent by the inspector to his next higher authority for keeping in safe custody for production in court, if required.

7. Sampling from the bulk fertilisers in ships, bulk carriers & bulk containers.

(i) Sampling equipments:-

(a) Sampling cup- The sampling cup

can be fabricated from non-corrosive metal. Inside dimensions of cup mouth may be  $3/4"$  x  $10"$  as per the diagram of the sampling cup given in Figure-1 below:-

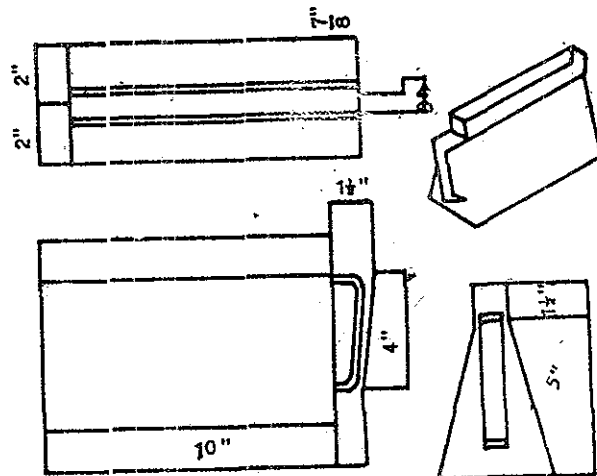


Figure 1 - Sampling cup

(b) Sampling probe for bulk fertiliser

The sampling probe should be made of non corrodable material such as stainless steel or brass. It may be a slotted double tube with solid cone tip having a length of about  $4\frac{1}{2}'$  -  $5'$  and diameter of about  $1\frac{1}{4}"$  to  $1\frac{1}{2}"$

(c) Scoop-Samples from the hatch can also be collected by a suitable scoop made of stainless steel or brass.

(ii) Sampling Procedure-

(a) Drawal of samples from the ship of bulk fertilisers-

A. Sampling from conveyor belt-

When the material is unloaded from the ships and transported to temporary/storage godowns through conveyor belts, the sample can be drawn by passing the sampling cup through the entire stream of material as the material drops from a transfer belt or spout. The long slot in the top of sampling cup should be perpendicular to the falling stream. Pass the cup through the complete stream at

an uniform speed, so that the cup will collect approximately equal amounts in each pass but will never over]

A minimum of 10 equally timed and speed stream must be taken during the transfer operation. However, the stream samples are not applicable unless uniform continuous flow of fertiliser is maintained for more than 3 minutes while lot is being sampled.

#### B. Sampling from hatch-

In case of bulk fertilisers, from each hatch, atleast 5-6 composite samples should be drawn from different depths and different points during unloading operation. To make one composite sample, minimum 5 samples should be drawn from different points at a specific depth. All these samples should be mixed to make one composite sample. The depths may be 0-5m, 5-10m, 10-15m, 15-20m and 20-25m depending upon the depth of hatch. The samples at a particular depth should be drawn after removal of material upto the required depth. The samples may be drawn with the help of sampling probe or scoop.

#### (b) Drawal of samples from Bulk Carriers- Trucks etc.-

The sample can be drawn as per vertical probing procedure of Association of Official Analytical Chemists. The sampling probe should be about 4½' to 5' length. Draw 10 vertical cuts from the following locations relative to the entire top of the conveyance. The 10 vertical cores are combined into a composite sample.

7	4	8
3	1 2	5
10	6	9

depth of the probe from the position indicated in the fig-2 and all cores are composited.

B.A ore sided or sloped pile may be sampled at the points illustrated in Fig.3 Withdraw one vertical core of material from location 1 & 6 and two cores at locations 2,3,4 & 5. Composite all the probe samples and prepare the composite sample for analysis as per procedure laid down in paras 1, 5 & 6.

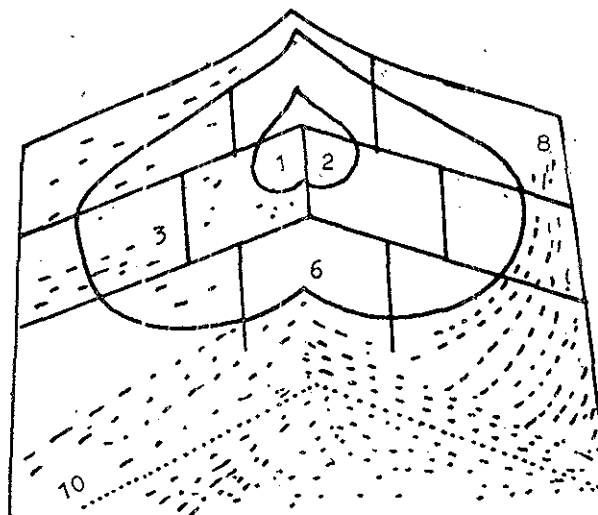


Figure 2—Sample points for coned or ridged pile.

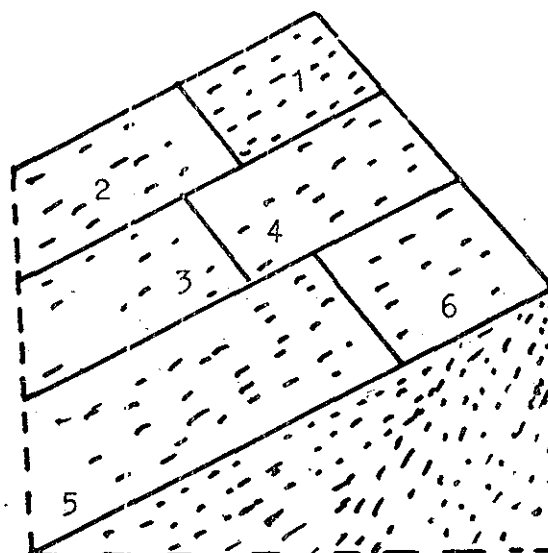


Figure 3—Sample points for ½ or one sided pile.

#### (c) Drawal of samples from bulk material in storage-

A. The bulk storage piles (level or flat) upto 100 tonnes could be sampled as per figure-2. Take 10 cores to the maximum possible

#### 8. Method of sampling of anhydrous ammonia-

##### (i) Scope

This method is for use in obtaining samples of anhydrous ammonia. The method is based on the assumption

that the material to be sampled is as claimed and contains only a small amount of impurity primarily water. It is recommended that duplicate samples be taken from each tank or vessel sampled.

(ii) Apparatus-

- Tubes for sampling tubes, heat resistant glass, conical centrifuge type 200 ml with lower 100 ml graduated from 0.2 ml in 0.05 ml divisions, 2-4 ml in 0.1 division 4-10 ml in 0.5 ml divisions and 10-100ml in 1.0 ml divisions. Tube type shown in Figure 5.
- Stoppers for tubes, rubber, with beat tube vent as in Figure 5.
- Sample carrier, constructed of plywood or aluminium as in Figure 5.
- Sampling line and connection assembly constructed as in Figure 4, with flexible steel sampling nose 48" long, 1/4" NPT coupling at each and 1/8" insulated steel tubing delivery tip at one end.
- Sample tube adapter, constructed from rubber stopper and 6 mm OD. glass or steel tubing as in figure 5.
- Protective equipment: Rubber or other non-porous gloves, offering complete protection to the hands and lower arms, full coverage goggles or approved gas mask.

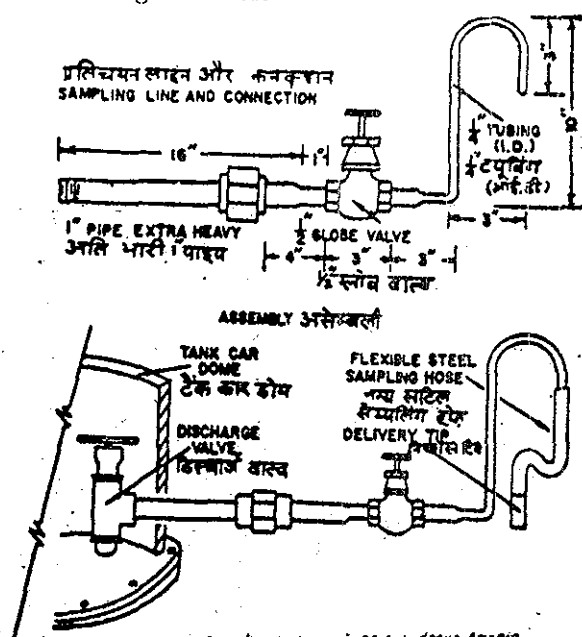


Fig. 4: Sampling & Analysis of Anhydrous Ammonia

SAMPLING AND ANALYSIS OF ANHYDROUS AMONIA

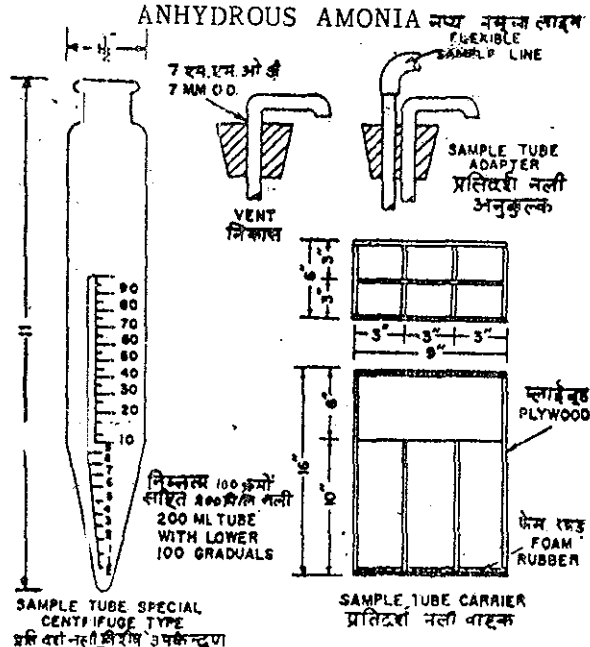


Figure 5

(iii) Reagents-

Charcoal, reagent, 14-20 mesh.

Note : If the sample is expected to contain excessive amount of water (one per cent or more), one piece of the charcoal may be added to each tube before introduction of the sample.

(iv) Procedure:-

- Place two dry, clean sampling tubes in the sample carrier.
- Connect the sampling line connection assembly to the unloading valve of the tank, vessel, or line to be sampled.
- Open the valve slowly and purge the sampling line connection assembly thoroughly by venting 3 to 4 litres of ammonia. Close the sample line globe valve.
- Remove the vented stoppers from the tubes and insert the adapter end of the sampling line connection assembly.
- Open the sample line valve and slowly fill the sampling tube to the 100 ml mark, close the sample line valve.
- Remove the sampling line adapter and insert the vented stopper in the sampling tube.
- Repeat steps, d and e and f and fill the second tube.
- Close tank discharge valve

and remove the sampling line connection assembly.

- (i) Either know or note the vessel, container, or line pressure of material sampled.
- (j) Tag the collected samples for identification and submit to the laboratory for processing by following methods in this section.

(v) Precaution

- (a) Liquid anhydrous ammonia causes severe burns on contact. It evaporates readily releasing the gas which may cause carrying degrees of irritation of the skin and mucous membranes and may injure severely the respiratory mucosa with possible fatal outcome.
- (b) Avoid contacting liquid ammonia. In case of contact, immediately flush the affected parts with plenty of water for atleast 15 minutes. Get medical attention at once in case of burns, especially to the eyes, nose and throat, or if the victim is unconscious.
- (c) Ammonia gas in concentrations of 6,000 to 10,000 ppm (by volume) is lethal within a few minutes. Irritation of the eyes, respiratory tract and throat results from concentrations as low as 500 to 1,000 ppm, a concentration of 2,000 ppm produces convulsive coughing and may be fatal after a short exposure i.e., less than half an hour. The maximum concentration tolerated by the skin for more than few seconds is 2 per cent (i.e. when suitable respiratory protection is worn). The maximum allowable concentration for 8 hour working exposure is 50 ppm. This is the least detectable order.
- (d) Obtain medical attention if exposure to the gas produce distress of any type.
- (e) Rubber or other non-porous gloves, offering complete protection to the hands and lower arms must be worn when sampling anhydrous ammonia. Full coverage goggles must also be worn to protect the eyes unless an approved gas mask is used. The gas mask need only to be used if sampling can not be done without possible inhalation of the vapours.

PART B.- METHODS OF ANALYSIS OF FERTILISERS

1. Preparation of sample for analysis in the laboratory

(i) Procedure

- (a) Reduce gross sample to quantity sufficient for analysis or grind not  $\geq 250$  gm of reduced sample without previous sieving.
- (b) For fertiliser materials and moist fertiliser mixtures, that form a paste on putting pressure, grind in porcelain pestle and mortar to pass sieve with 1 mm circular openings or No.20 standard sieve.
- (c) For dry mixtures that tend to segregate, grind in a porcelain pestle and mortar to pass No. 40 standard sieve.
- (d) Grind as rapidly as possible to avoid loss or gain of moisture during operation.
- (e) Mix thoroughly and store in tightly stoppered bottles.

2. Determination of moisture

(Not applicable to samples that yield volatile substances other than water at drying temperature).

(i) Procedure

- (a) Weight to the nearest mg about 2 gm of the prepared sample in a weighed, clean, dry squat form weighing bottle.
- (b) Heat in an oven for about 5 hours at 99-101°C to constant weight. Cool in a desicator and weigh.
- (c) In case of sodium nitrate, ammonium sulphate and potassium salts heat to constant weight at 129-131°C.
- (d) Report percentage loss in weight as moisture at temperature used.

Calculations

Free moisture per cent by weight =  $\frac{100(B-C)}{B-A}$

- A = Weight of the bottle.
- B = Weight of the bottle plus material before drying.
- C = Weight of the bottle plus material after drying.

(Reference "Methods of Analysis".  
AOAC. 1965)

(ii) Moisture in Ammonium Chloride.

(a) Weigh accurately about 5 gm of prepared sample in a weighed shallow porcelain dish and dry for 24 hours in a vacuum desiccator over sulphuric acid and re-weigh.

(b) Preserve the dried material for subsequent tests.

Calculations

$$\text{Moisture per cent by weight} = 100 \times \frac{W_1}{W_2}$$

$W_1$  = loss in weight in gm on drying, and

$W_2$  = weight in gm of the prepared sample taken for the test.

(iii) Samples like urea, diammonium phosphate and ammonium nitrate which yield volatile substances other than water at drying temperature, the Karl Fischer method given below is used for the determination of moisture.

(iv) Reagents

(a) Iodine solution - Add 125gm of iodine to a mixture of 650 ml of methanol and 200 ml of pyridine contained in a flask, and immediately close the flask tightly.

(b) Sulphur dioxide solution- Pass dry sulphur dioxide into 100 ml of pyridine contained in a 250 ml graduated cylinder and cooled in an ice bath, until the volume reaches 200 ml.

(c) Fischer Reagent - Slowly add iodine solution to the cooled sulphur dioxide solution stopper immediately and shake well until the iodine is dissolved. Transfer the solution to an automatic pipette, protected from absorption of moisture by a drying agent and allow to stand for 24 hours before standardising. The reagent deteriorates continuously and it should be standardized within one hour before use.

(d) Standard water solution - Measure exactly 2 ml of water into a thoroughly dry 1 litre volumetric flask, dilute to volume with methanol. Retain sufficient quantity of the same methanol for a blank determination. Keep the solution in tightly closed containers.

(v) Procedure

(a) Determination of end point in Karl Fischer Titration - In many cases the end point can be detected visually by the change of colour from a light brownish yellow to amber. But when the end point is not clearly defined, the electrometric method for determining the end point should be adopted. Adjust the potentiometer so that when a small excess (0.02ml) of the reagent is present, a current of 50 to 150 microamperes is recorded. The solution should be continuously and vigorously stirred. At the beginning of the titration, a current of only a few microamperes will flow. After each addition of reagent, the pointer of the micrometer deflected but rapidly returns to the original position. At the end point a deflection is obtained which endures for a longer period.

(b) Standardization of the Fischer reagent - Pipette exactly 10ml of methanol into a dry titration flask and titrate with the Fischer reagent to the end point ( $V_1$ ). Pipette exactly 10ml of the standard water solution into the flask and titrate to the end point ( $V_2$ ).

(c) Titration of the material - Transfer 25 ml. of methanol to the titration flask and titrate to the end point ( $V_3$ ) with the Fischer reagent. Do not record the volume consumed. Quickly transfer to the titrated liquid an accurately weighed quantity of the material containing 10 to 50 mg. of water. Water stir vigorously and titrate to the end point.

$$\text{Calculation} = \frac{0.1 \times W(V_3 - 2.5V_1)}{(V_2 - V_1) A}$$

Moisture per cent by weight

where,

$W$  = Weight in mg of water contained in 10 ml of standard water solution

$V_3$  = Total volume in ml. of the reagent used in titration in (c).

$V_1$  = volume in ml. of reagent used in titration of methanol in (b), and



$V_2$  = total volume in ml. of the reagent used in titration in (b).

A = weight in gm. of the material taken for the test in (c).

(Reference IS. Specification for Urea, technical and pure, IS. 1781-1961)

### 3. Determination of nitrogen

Methods of determination of total nitrogen, ammoniacal nitrogen, nitrate nitrogen, and urea nitrogen have been described separately in this section. These methods can be adopted both for straight and mixed fertilisers. Scope of each method with various combinations have also been described with each method.

The relevant methods of analysis which have been described are as given below:

- (a) Total Nitrogen - for nitrate free samples.
- (b) Total Nitrogen - for nitrate containing samples.
- (c) Total Nitrogen - for materials with high Cl:  $\text{NO}_3$  ratio and to materials containing only water soluble nitrogen.
- (d) Determination of ammoniacal nitrogen.
- (e) Determination of ammoniacal and nitrate nitrogen.
- (f) Determination of nitrate nitrogen.
- (g) Determination of water insoluble nitrogen.
- (h) Determination of urea nitrogen.

#### (i) Detection of nitrate

For adopting a specific method as described above, it is necessary to detect the presence of nitrates in the sample before a particular method is adopted. The procedure for detection of nitrates is given below:-

- (a) Mix 5 gm sample with 25 ml. hot water and filter.
- (b) To one volume of this solution add 2 volumes of sulphuric acid, free from  $\text{HNO}_3$  and oxides of N, and let it cool.
- (c) Add a few drops of concentrated  $\text{FeSO}_4$  solution in such a manner that fluids do not mix.
- (d) If the nitrates are present junction shows at first purple, afterward brown, or if only minute quantity

is present, reddish colour.

- (e) To another portion of solution add 1 ml. 1 per cent  $\text{N}_2\text{NO}_3$  and test as before to determine whether enough  $\text{H}_2\text{SO}_4$  was added in the first test.

(Reference "Methods of Analysis" AOAC, 1965)

#### (ii) Reagents for determination of total nitrogen

- (a) Sulphuric acid - 93-98 per. cent  $\text{H}_2\text{SO}_4$ , N free.
- (b) Copper sulphate -  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  reagent grade, N free
- (c) Potassium sulphate (or anhydrous sodium sulphate) - reagent grade.
- (d) Salicylic acid - reagent grade, N free.
- (e) Sulphide or thiousulphate solution- Dissolve 40 gm commercial  $\text{K}_2\text{S}$  in 1 litre distilled water.

(Solution - of 40 gm  $\text{Na}_2\text{S}$  or 80 gm  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in 1 litre may be used)

- (f) Sodium hydroxide - Pellets or solution, nitrate free. For solution dissolve approximately 450 gm solid NaOH in distilled water and dilute to 1 litre (Sp. gr. of solution should be 1.36 or higher).
- (g) Zinc granule - reagent grade.
- (h) Zinc dust - Impalpable powder.
- (i) Methyl red indicator - Dissolve 1 gm. methyl red in 200 ml. alcohol.
- (j) Hydrochloric or sulphuric acid standard solution, - 0.5N or 0.1N when amount of N is small.
- (k) Sodium hydroxide standard solution - 0.1N (or other specified concentration).
- (1) Standardize each standard solution with primary standard and check one against another.
- (2) Test reagents before using, by blank determination with 2 gm. sugar which insures partial reduction of any nitrates present.

Caution: Use freshly opened sulphuric acid or add dry  $\text{P}_2\text{O}_5$  to avoid hydrolysis of nitrites and Cyanates. Ratio of salt to acid

(wt: Vol) should be approximately 1:1 at end of digestion for proper temperature control. Digestion may be incomplete at lower ratio, N may be lost at higher ratio.

(iii) Apparatus

(a) For digestion - Use Kjeldahl flask of hard moderately thick, well annealed glass with total capacity approximately 500-800 ml. Conduct digestion over heating device, adjusted to bring 250 ml. water at 25°C to rolling boil in approximately for 5 minutes. Add 3-4 boiling chips to prevent superheating.

(b) For distillation - Use Kjeldahl or other suitable flask of 500-800 ml. capacity filled with rubber stopper through which passes lower end of efficient scrubber bulb or trap to prevent mechanical carryover of NaOH during distillation. Connect upper end of bulb tube to condenser tube by rubber tubing. Trap outlet of condenser in such a way as to insure complete absorption of ammonia distilled over into acid in receiver.

(iv) Total nitrogen (in nitrate free samples)

(a) Procedure

- (1) Place weighed sample (0.7- 2.2 gm) in digestion flask.
- (2) Add 0.7 gm. copper sulphate, 15 gm. powdered  $K_2SO_4$  or anhydrous  $Na_2SO_4$  and 25 ml.  $H_2SO_4$ .
- (3) If sample more than 2.2 gm. is used, increase sulphuric acid by 10 ml. for each gm. sample.
- (4) Place flask in inclined position and heat gently until frothing ceases (if necessary add small amount of Paraffin to reduce frothing).
- (5) Boil briskly until solution clears and then for at least 30 minutes longer (2 hours for sample containing organic material).
- (6) Cool add approximately 200 ml. distilled water, cool below 25°C.
- (7) Add to the flask a layer of sodium hydroxide (25 gm. solid reagent or enough solution to make contents strongly alkaline) without agitation.
- (8) Immediately connect flask to

distilling bulb or condenser and with tip of condenser immersed in standard acid in receiver.

- (9) Rotate flask to mix contents thoroughly, then heat until all ammonia has distilled (at least 150 ml. distillate).
- (10) Titrate excess standard acid in distillate with standard sodium hydroxide solution, using methyl red as an indicator.
- (11) Correct for blank determination on reagents.

Calculation

$$\text{percent Nitrogen} = \frac{(A - B) \times 140}{W}$$

A = Ml. of standard acid used.

B = ml. of standard NaOH used.

Na = Normality of standard acid.

Normality of standard NaOH.

weight of the sample taken in grams.

(Reference "Methods of Analysis", A.O.A.C. 1965):

- (v) Total Nitrogen (for nitrate containing samples)  
(Not applicable to samples containing high concentrations of nitrate nitrogen and chlorides)

(a) Procedure

- (1) Place weighed sample (0.7- 2.2 gm) in digestion flask.
- (2) Add 40 ml.  $H_2SO_4$  containing 2 grams salicylic acid. Shake until thoroughly mixed and let stand, with occasional shaking, 30 minutes or more.
- (3) Then add (i) 5 grams  $Na_2S_2O_4 \cdot 5H_2O$  or (ii) 2 grams zinc dust (as impalpable powder not granulated zinc or filings).
- (4) Shake the flask and let it stand for five minutes, then heat over low flame until frothing ceases.
- (5) Turn off heat, add 0.7 grams copper sulphate, 15 gm. powdered  $K_2SO_4$  (or anhydrous  $Na_2SO_4$ ), and boil briskly until solution clears, then at least 30 minutes longer (2 hours for samples containing organic material).

Proceed further as in 6-11 of 3 (iv).

Calculations

Same as in 3 (iv)

(Reference "Methods of Analysis".- A.O.A.C. 1965).

(vi) Total nitrogen (for materials with high Cl : NO<sub>3</sub> ratio and to materials containing only water soluble nitrogen)

(a) Reagents

Reduced iron powder, electrically reduced, N.F. (National Formularity). For other reagents see 3(ii).

(b) Procedure

Mixed Fertilisers

(1) Place 0.5-2.0 gm. sample in Kjeldahl flask and add 2.5 grams reduced Fe (5 gms. is enough for 0.185 grams. NO<sub>3</sub>).

(2) Add approximately 25 ml. distilled water, rotating flask at angle to wash down the sample.

(3) Let it stand for 15 minutes with occasional agitation, to insure complete solution of all soluble salts.

(4) While rotating flask add 25 ml. cold H<sub>2</sub>SO<sub>4</sub> (1+1) and let it stand until visible reaction ceases. (use hood or vented digestion unit).

(5) Add boiling chips and boil 15-20 minutes, but do not take to dryness. (For samples containing organic matter, use 50 ml. cold H<sub>2</sub>SO<sub>4</sub> (1+1), boil for 15-20 minutes, add 0.7 gm. HgO add heat again for 30 minutes). Cool and proceed further as in (6)-(11) of 3 (iv).

Calculations

Same as in 3 (iv)

(Reference "Methods of Analysis". A.O.A.C., 1965).

(vii) Determination of ammoniacal nitrogen

(Distillation Method).

(This method is for the determination if nitrogen present or available in the sample as ammonium ion. This method assumes that Urea is absent from the sample. Not applicable to MgNH<sub>4</sub> PO<sub>4</sub> and Fe NH<sub>4</sub> PO<sub>4</sub>).

(a) Procedure

(1) Place 0.7-3.5. grams; according to NH<sub>3</sub> content of the sample, in distillation flask with approximately 300 ml. water and 2 grams of freshly ignited carbonate free Mgo or NaOH solution.

(2) Connect the flask to condenser by Kjeldahl connecting bulb.

(3) Distil 100 ml. liquid into measured quantity of standard acid and titrate with standard NaOH solution, using methyl red as an indicator.

Calculations

$$\text{Percent Nitrogen} = \frac{(ANa - BNb) \times 0.01401 \times 100}{W}$$

A = ml. of standard acid used.

B = ml. of standard NaOH.

Na = normality of acid.

Nb = normality of NaOH.

W = weight of the sample taken in gms.

(Reference-Recommended analytical methods of the National Plant Food Institute, Washington, D.C., 1961).

(viii) Determination of ammoniacal and nitrate nitrogen (Devarda Method)

(This method is for the determination of total nitrogen when only nitrate or mixture of nitrate and ammoniacal nitrogen is present. This method assumes the absence of Urea, Calcium Cyanamide and organic matter from the sample).

(a) Procedure

(1) Place 0.35-0.5 gms. sample in 600-700 ml. flask and add 300 ml. water, 3 grams Devarda Alloy, and 5 ml. NaOH solution (42 percent by weight pouring later down side of flask so that it does not mix at once with contents).

(2) Allow the flask to stand for 15 minutes.

(3) By means of Division (j. Ind. Eng. Chem. 11, 465 (1919) or other suitable scrubbing bulb that will prevent passing over of any spray connect with condenser.

tip of. hich always extends beneath surface of standard acid in receiving flask.

- (4) Mix contents of distilling flask by rotating.
- (5) Heat slowly at first, and then at a rate to yield 250 ml. distillate in 1 hour.
- (6) Collect distillate in measured quantity of standard acid and titrate with standard NaOH solution, using methyl red as an indicator.
- (7) In analysis of nitrate salts dissolve 3.5 or 5.0 grams in water, to 250 ml. and use 25 ml. aliquot.

#### Calculations

Same as in 3 (iv)

(Reference "Methods of Analysis". A.O.A.C. 1965).

(ix) Determination of nitrate nitrogen

(Applicable in presence of calcium cyanamide and urea in the mixture).

#### (a) Procedure

- (1) Determine total nitrogen as in 3 (v)
- (2) Determine water insoluble N as in 3 (x) but use 2.5 grams sample. Dilute to 250 ml.
- (3) Determine ammoniacal N in 50 ml filtrate as in 3 (vii)
- (4) Place another 50 ml. portion filtrate in 500 ml. Kjeldahl flask and 2 grams.  $\text{Fe SO}_4 \cdot 7\text{H}_2\text{O}$  and 20 ml.  $\text{H}_2\text{SO}_4$ . (If total N is 5 percent use 5 gm.  $\text{Fe SO}_4 \cdot 7\text{H}_2\text{O}$ ). Digest over hot flame until all water is evaporated and white fumes appear and continue digestion at least ten minutes to drive off Nitrate N. If severe bumping occurs, add 10-15 glass beads. Add 0.65 grams Hg or 0.7 grams  $\text{HgO}$  and digest until all organic matter is oxidized cool, dilute, add the  $\text{K}_2\text{S}$  solution and complete determination as 3 (iv). Before distillation add pinch of mixture of Zinc dust and granular

"20 mesh", Zinc to each flask to prevent bumping.

#### Calculations

Total N (a)-water insoluble N (b)-water soluble N. Water Sol N-N obtained in (d)-Nitrate N.

(Reference Methods of Analysis A.O.A.C., 1965).

(X) Determination of water insoluble nitrogen.

#### (a) Procedure

- (1) Place 1 or 1.4 grams sample in 50 ml. baker, wet with alcohol.
- (2) Add 20 ml. water and let it stand for 15 minutes stirring occasionally.
- (3) Transfer supernatant liquid to 11 cm. Whatman No.42 paper to 60° long stem funnel 2.5" diameter and wash residue 4 or 5 times by decanting with water at room temperature (20-25°C).
- (4) Finally, transfer all residue to filter and complete washing until filtrate measures 250 ml.
- (5) Determine N as in 3 (iv).

(xi.) Determination of Urea nitrogen.

(This method is for the determination of urea content of any mixed fertilisers).

#### (a) Reagents

(1) Neutral urease solution- shake 1 gm. jack bean meal with 100 ml. water for 5 minutes. Transfer 10 ml. solution to 250 ml. (Erlenmeyer flask, dilute with 50 ml. water, and add 4 drops methyl purple indicator. Titrate with 0.1 N HCl to reddish purple, then back titrate to green with 0.1 N NaOH. From difference in ml. calculate amount of 0.1 N HCl required to neutralize remainder of solution (usually approximately 2.5 ml. per 100 ml.) add this amount of acid and shake well.

#### (b) Procedure

- (1) Weight  $10 \pm 0.01$  gm. sample and transfer to 15 cm. Whatman No.12 fluted filter paper.
- (2) Leach with approximately 300 ml. water into 500 ml. volumetric flask.
- (3) Add 75-100 ml. saturated barium

hydroxide solution to precipitate phosphates.

(4) Let it settle and test for complete precipitation with few drops of saturated barium hydroxide solution.

(5) Add 20 ml. 10 per cent sodium carbonate solution to precipitate excess barium and any soluble calcium salts.

(6) Let it settle and test for complete precipitation.

(7) Dilute to volume, mix and filter through 15 cm. Whatman No. 12 fluted paper.

(8) Transfer 50 ml. aliquot (equivalent to 1 gm. sample) to 200 or 250 ml. Frlenneyer flask and add 1 to 2 drops methyl 1 purple indicator.

(9) Acidify solution with 2N HCl and add 2 to 3 drops excess.

(10) Neutralize solution with 0.1 N NaOH to first change in colour indicator.

(11) Add 20 ml. neutral ureas solution, close flask with rubber stopper and let it stand for 1 hour at 20-25°C.

(12) Cool the flask in ice water slurry and titrate at once with 0.1 N HCl to full purple colour, then add approximately 5 ml. excess.

(13) record total volume added, back titrate excess HCl with 0.1 N NaOH to neutral end point.

#### Calculations

Percentage urea =  $\frac{(\text{ml. } 0.1 \text{ NHCl} - \text{ml. } 0.1 \text{ N NaOH}) \times 0.3003 \text{ wt. of sample}}{\text{sample}}$

(Reference "Methods of Analysis" A.O.A.C. 1965).

(xii) Determination of biuret.

#### (a) Reagents

(1) Alkaline tartarate solution - Dissolve 40 gm. NaOH in 50 ml. water cool, add 50 gm. of  $\text{NaKC}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O}$  and dilute to 1 litre. Let it stand for one day before use.

(2) Copper sulphate solution - Dissolve 15 gm.  $\text{CuSO}_4 \cdot \text{H}_2\text{O}$  in  $\text{CO}_2$  free water and dilute to 1 litre.

(3) Biuret standard solution - 1 mg./ml.

Dissolve 100 mg reagent grade biuret in  $\text{CO}_2$  free water and dilute to 100 ml.

(4) Ion exchange resin. Fill 50 ml. burette with 30 cm. column of Amberlite IR 120(H) resin on glass wool plug. Regenerate column after each use by passing 100 ml.  $\text{H}_2\text{SO}_4$  (1+9) or HCl (1+4) through the column approximately 5 ml. per minute and then washing with water until pH of effluent is 6.0.

#### (b) Preparation of standard curve.

(1) Transfer series of aliquots, 2-50 ml. of standard biuret solution to 100 ml. volumetric flask.

(2) Adjust volume to approximately 50 ml. with  $\text{CO}_2$  free water. Add one drop of methyl red and neutralize with 0.1N  $\text{H}_2\text{SO}_4$  to pink colour.

(3) Add with swirling 20 ml. alkaline tartarate solution and then 20 ml.  $\text{CuSO}_4$  solution.

(4) Dilute to volume. Shake for 10 seconds and place in water bath for 15 minutes at  $30 \pm 0.5^\circ\text{C}$ .

(5) Also prepare reagent blank.

(6) Determine absorbance of each solution against blank at 555 mμ (instrument with 500 - 570 mμ filter is also satisfactory) with 2-4 cm. cell, and plot standard curve.

#### (c) Procedure

##### A. In urea.

(1) Stir continuously 2-5 gm sample in 100 ml. approximately  $50^\circ\text{C}$  water for 30 minutes.

(2) Filter and wash into 250 ml. volumetric flask and dilute to volume.

(3) Transfer 25 ml. aliquot to 100 ml. volumetric flask and proceed as given under preparation of standard curve 3(xii).

##### B. In mixed fertilisers

(1) Stir continuously 10-20 gm. sample in 150 ml. approximately ( $50^\circ\text{C}$ ) hot water for 30 minutes.

(2) Filter and wash into 250 ml. volumetric flask and dilute to volume.

(3) Transfer 25 ml. aliquot to column 3(xii)(a)(4) and adjust flow to 4-5 ml./minute.

(4) Receive eluate in 100 ml. beaker.

(5) When liquid level falls to top of resin bed, wash with two 25 ml. portion water.

(6) To eluate and washings add two drops of methyl red and then 0.1N NaOH to yellow colour.

(7) Add 0.1N  $\text{H}_2\text{SO}_4$  until solution just turns pink.

(8) Transfer to 100 ml. volumetric flask and dilute to volume with  $\text{CO}_2$  free water.

(9) Transfer 50 ml. aliquot to 100 ml. volumetric flask and proceed as in preparation of standard curve given above.

#### Calculations

From standard curve determine concentration of biuret in final dilution, then per cent of

$$\text{biuret} = \frac{C \times 100}{W}$$

where C = Concentration in mg/ml of biuret in final dilution obtained from standard curve.

W = Concentration of original sample in final dilution expressed as mg/ml.

(Reference "Methods of Analysis"  
A.O.A.C. 1965)

(xiii) Determination of free acidity in ammonium sulphate (as  $\text{H}_2\text{SO}_4$ )

#### (a) Reagents

(1) Standard sodium hydroxide solution- 0.02N.

(2) Methyl red indicator - Dissolve 0.15 gm. of water soluble methyl red in 500 ml. water.

(3) Methyl red - Methyl blue mixed indicator solution - prepared by mixing equal volumes of 0.2 per cent solution in rectified spirit of methyl red and 0.1 per cent solution in rectified spirit of methylene blue.

#### (b) Procedure

(1) Dissolve about 20 gm. of prepared sample, accurately weighed in about 50 ml. cold natural water.

(2) Filter and make up the volume to about 200 ml.

(3) Titrate with standard sodium hydro-

xide solution, using one or two drops of methyl red as indicator.

(4) If satisfactory end point with methyl red is not obtained, methylene red-methylene blue mixed indicator may be used.

(5) Use preferably a micro biuret for this titration.

The filtering medium shall be neutral and shall not contain any alkaline material which would neutralize free acid.

#### Calculations

Free acidity as  $\text{H}_2\text{SO}_4$  per cent  $\frac{4.904 \text{ AN}}{W}$

A = Volume of ml. of standard NaOH solution.

N = Normality of standard NaOH solution.

W = Weight in gm. of prepared sample taken for the test.

(Reference-Specifications for ammonium sulphate, fertilizer grade IS:826-1967)

(xiv) Determination of Arsenic in Ammonium sulphate ( $\text{As}_2\text{O}_3$ ).

#### (a) Reagents

(1) Lead acetate solution- Prepare 10 per cent solution of lead acetate with sufficient acetic acid added to clear the solution.

(2) Dry lead acetate paper- Cut filter paper (Whatman No.1 or equivalent) into strips 70x50 mm and keep them permanently suspended in lead acetate solution in a glass stoppered bottle. Before use, take out the strips and dry them in an atmosphere free from hydrogen sulphide.

(3) Mercuric Bromide solution- Dissolve 5 gm of mercuric bromide in 100 ml. rectified spirit.

(4) Sensitized Mercuric Bromide paper strips-cut filter paper (Whatman No.1 or equivalent) into strips 120x2.5 mm. Keep the strips permanently suspended in dark in a glass stoppered cylinder or amber bottle having mercuric bromide solution. Before use take out a strip, press it between sheets of filter paper and dry it in an atmosphere free from hydrogen sulphide.

(5) Dilute sulphuric acid-approximately 5 N.

(6) Concentrated hydrochloric acid.

(7) Potassium iodine solution- 15 per cent.

(8) Stannous chloride solution- Dissolve 80 gm. of stannous chloride in 100 ml. water containing 5 ml. of concentrated hydrochloric acid. If the solution is turbid add a few more ml. of hydrochloric acid and boil until clear solution is obtained. Add some metallic tin to the solution to prevent oxidation.

(9) Zinc- It is recommended that zinc rods prepared as described below should be used for routine work, however, pellets described may be used.

(b) Preparation of zinc rods.

(1) Take a clean and dry hard glass test tube of 10 mm internal diameter and 20 cm. length.

(2) Heat the test tube over a flame of Bunsen or blow pipe burner and add slowly arsenic free granulated zinc in small portions (1 to 2 gm at a time) the next portion being added after the first one has completely melted.

(3) Continue heating and adding zinc until the melt is about 10 cm. high.

(4) Heat the clean melt for half an hour and then cool to room temperature.

(5) Break the tube to obtain the rod of zinc.

(6) Cut rod into pieces 20 mm. long.

(7) Coat the plane ends of the pieces with a paste of magnesium carbonate and gum arabic solution and dry.

(8) Coat the pieces all over with 1.5 mm. thick layer of paraffin wax.

(9) When required for use, scrap off the wax from the plane ends with a knife protecting wax colour round the rods.

(10) Remove the paste from the plane ends by soaking in water and activate the exposed surface by dipping in a solution containing one part of stannous chloride solution and seven parts of concentrated hydrochloric acid.

(c) Preparation of zinc pellets

Treat zinc shots passing through IS sieve 570 (aperture 5660 microns) and retained on IS sieve 280 (aperture 2818 microns) with concentrated hydrochloric acid until the surface of zinc becomes clean and dull. Weigh and keep under water, preventing contamination with dust.

(d) Standard sodium hydroxide solution- approximately 20 per cent.

(e) Standard arsenic trioxide solution.

(1) Dissolve 1.0 gm. of resublimed arsenic trioxide ( $As_2O_3$ ) in 25 ml. sodium hydroxide solution and neutralize with dilute sulphuric acid.

(2) Dilute with freshly distilled water containing 10 ml. of concentrated sulphuric acid per litre and make up the volume to 1 litre.

(3) Again dilute 10 ml. of this solution to 1 litre with water containing sulphuric acid and finally dilute 100 ml. of this solution to 1 litre with water containing sulphuric acid. One ml. of this solution contains 0.001 mg. of arsenic trioxide ( $As_2O_3$ ). The dilute solution shall be prepared freshly when required.

(f) Procedure

(1) Dissolve 1.0 gm of the prepared sample in 20 ml. water.

(2) Place dry lead acetate paper in the lower portion of the tube B (in figure-6) and glass wool moistened with lead acetate solution in its proper portion.

(3) Place the sensitized strips of mercuric bromide paper in tube A and connect the tubes together with a rubber stopper.

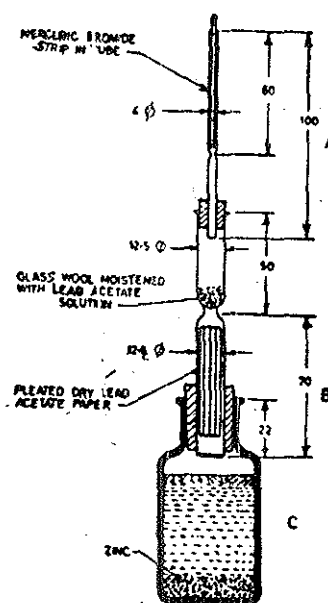


Figure 6—Modified Gutzeit Method of test for arsenic.

(4) Introduce the solution of the material into the bottle C (120 ml.) and then add 10 ml of dilute sulphuric acid. Add 0.5 ml. of stannous chloride solution 5 ml. potassium iodide solution and make up the volume with water to about 50 ml.

(5) Mix the contents and drop about 10 gm. of zinc. Immediately fit in position the rubber stopper carrying the tube. B.

(6) Place the bottle in a warm place at about 40°C.

(7) At the end of two hours remove the test strip by means of tweezers.

(8) Carry out the test prescribed above using a volume of standard arsenic trioxide solution containing 0.1 mg of arsenic trioxide, in place of the solution of the material and compare the strain produced with the material with that produced with arsenic trioxide solution.

(9) The limit prescribed in the material specification shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with the material is not greater than those produced with the arsenic solution.

(Reference "Modified Gutzet method of test for arsenic" IS-2088-1962)

(xv) Determination of calcium nitrate (for calcium ammonium nitrate and nitrophosphates).

(a) Reagents

(1) N = Amyl Alcohol

(2) Dilute hydrochloric acid- approximately 4 N.

(3) Standard calcium solution-weight 1.0 gm of calcium carbonate dried at 120° ± 5°C and dissolve in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask.

(4) Ammonium chloride-ammonium hydroxide buffer solution. Dissolve 67.5 gm. ammonium chloride in a mixture of 570 ml. of ammonium hydroxide (sp.gr.0.92) and 250 ml. water. Also dissolve separately a mixture of 0.931 gm of disodium ethylene diamine tetra-acetate dihydrate and 0.616 gm of magnesium sulphate ( $MgSO_4 \cdot 7H_2O$ ) in about 50 ml. of water. Mix the two solutions and dilute to 1 litre.

(5) Standard disodium ethylene diamine Tetra-acetate (EDTA) solution-weigh 3.72 gm. of disodium ethylene diamine tetra-acetate dihydrate in water, and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against standard calcium solution following the procedure given below.

(6) Eriochrome Black T Indicator solution- Dissolve 0.1 gm in 20 ml of rectified spirit. The solution shall be used for not more than a week.

(b) Procedure

(1) Grind quickly about 5 gm. of the material, accurately weighed, with about 50 ml. of amyl alcohol in a pestle and mortar and transfer the contents to a conical flask.

(2) Wash the pestle and mortar with a few ml. of amyl alcohol and add the washings to the flask.

(3) Shake the contents of the flask manually or in a mechanical shaker for about half an hour and then filter.

(4) Transfer the filtrate to a separating funnel and extract calcium nitrate completely with water in five to six instalments.

(5) A few drops of dilute hydrochloric acid may be added during the extraction with water to avoid formation of an emulsion of amyl alcohol with water.

(6) Concentrate the water extract at low temperature to nearly half its volume.

(7) Transfer the concentrated solution to a conical flask, add 5 ml. of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution to a pure blue end point.

Calculations

$$\text{Calcium nitrate per cent by weight} = \frac{8.2 \text{ NV}}{W}$$

Where N= Normality of standard EDTA solution.

V= Volume in ml. of standard EDTA solution used in the titration, and

W= Weight in gm. of the material taken for test.

(Reference: IS specifications of CAN IS: 2409-1963)

(xvi) Determination of Chlorides other than ammonium chloride.

(a) Reagents

(1) Standard silver nitrate solution-0.1 N

(2) Concentrated nitric acid-conforming to IS: 264-1950.

(3) Ferrous ammonium sulphate solution saturated in water and stabilized by addition of 50 ml. nitric acid.

(4) Standard Ammonium thiocyanate solution-0.1.N.

(b) Procedure

(1) Dissolve about 0.2 gm. of the prepared sample, previously dried as in procedure



for determination of moisture and accurately weighed, in about 40 ml water.

(2) Add exactly 50 ml. of standard silver nitrate solution and 5 ml. of concentrated nitric acid.

(3) Add 0.5 ml. of nitrobenzene and make up the volume of the mixture to exactly 100 ml. with water.

(4) Take exactly 50 ml. of the solution and add 2 ml. of ferric ammonium sulphate solution.

(5) Titrate the excess of silver nitrate in this portion with standard ammonium thiocyanate solution.

(6) Carry out a blank test following the procedure given as above but without using the material.

#### Calculations

$$\text{Total chlorides (as Cl) per cent by weight (on dry basis)} = \frac{7.07(V_1 - V_2)N}{W} \times 100$$

Where

$V_1$  = Volume in ml. of standard ammonium thiocyanate used in the blank determination.

$V_2$  = Volume in ml. of standard ammonium thiocyanate solution used in the test with the material.

$N$  = Normality of standard ammonium thiocyanate solution.

$W$  = Weight in gm. of the dried prepared sample taken for the test.

Express the ammoniacal nitrogen content per cent by weight of the material determined earlier in terms of Cl as follows:

Chloride equivalent of the ammoniacal nitrogen content, per cent by weight =  $2.531 \times A \dots Y$  where  $A$  is the ammoniacal nitrogen content determined earlier.

Balanced chloride equivalent to sodium chloride (NaCl) per cent by weight =  $1.648 \times (X - Y)$ .  $(X - Y)$  = Balanced chlorides other than ammonium chloride.

(Reference IS Specification for ammonium chloride fertiliser grade (Revised) IS: 1114-1964 calculations modified).

#### 4. Determination of phosphates.

Methods of determination of total phosphates, water soluble phosphates, citrate soluble phosphates, citrate

insoluble phosphates and citric acid soluble phosphates have been described separately in this section. These methods are applicable to straight as well as mixed phosphatic fertilisers.

(i) Preparation of solution of the samples.

(Separate methods of preparing solutions of the samples have been described according to the nature of the sample).

#### (a) Reagent

Magnesium nitrate solution - Dissolve 950 gm. P-free  $Mg(NO_3)_2 \cdot 6H_2O$  in water and dilute to 1 litre.

#### (b) Procedure

(1) Treat 1 gm sample by (A), (B), (C), (D) or (E) method depending on the nature of the sample.

(2) Cool solution transfer to 200 to 250 ml. volumetric flask, dilute to volume, mix and filter through dry filter.

A. (Suitable for materials containing small quantities of organic matter). Dissolve in 30 ml.  $HNO_3$  and 3-5 ml.  $HCl$  and boil until organic matter is destroyed.

B. (Suitable for fertilisers containing much Fe or Al phosphates and basic slag). Dissolve in 15-30 ml.  $HCl$  and 3-10 ml.  $HNO_3$ .

C. (Suitable for organic material like cottonseed meal alone or in mixture). Evaporate with 5 ml. of the  $Mg(NO_3)_2$  solution, ignite and dissolve in  $HCl$ .

D. (Generally applicable to materials or mixtures containing large quantities of organic matter). Boil with 20-30 ml.  $H_2SO_4$  in 200 ml. flask adding 2-4 gm of  $N_2O_5$  or  $KNO_3$  at beginning of digestion and small quantity after solution is nearly colourless, or adding the nitrate in small portions from time to time. When solution is colourless, cool add 150 ml. water and boil for few minutes. Before adding  $N_2O_5$  or  $KNO_3$ , let mixture digest, at gentle heat if necessary, until violence of reaction is over.

E. (Suitable for all fertilizers). Boil gently for 30-45 minutes with 20-30 ml  $HNO_3$  in a suitable flask (Preferably a Kjeldahl for samples containing large quantities of organic matter to oxidize all easily oxidizable matter). Cool and add 10-20 ml. of

70-72 per cent perchloric acid. Boil very gently until solution is colourless or nearly so and white dense fumes appear in flask. Do not boil to dryness at any time (Danger). (With samples containing large quantities of organic matter temperature should be raised to fuming point approximately 170°C, over a period of 1 hour at least). Cool slightly, add 50 ml. water and boil for few minutes.

(ii) Gravimetric Quinoline Molybdate method for determination of total phosphorus.

#### (a) Reagents

(1) Citric molybdic acid reagent- Dissolve 54 gm. 100 per cent molybdic anhydride ( $MgO_3$ ) and 12 gm. NaOH with stirring in 400 ml. hot water and cool. Dissolve 60 gm. citric in mixture of 140 ml. HCl and 300 ml. water and cool. Gradually add molybdic solution to citric acid solution with stirring. Cool, filter and dilute to 1 litre. (Solution may be green or blue, colour depends on exposure to light). If necessary add 0.5 per cent  $KBrO_3$  solution drop by drop until green colour becomes pale. Store in dark in polyethylene bottle.

(2) Quinoline solution - Dissolve 50 ml. synthetic quinoline with stirring in mixture of 60 ml. HCl and 300 ml. water. Cool dilute to 1 litre, and filter. Store in polyethylene bottle.

(3) Quimociac reagent - Dissolve 70 gm. of sodium molybdate dihydrate in 150 ml. water. Dissolve 60 gm. citric acid in mixture of 85 ml.  $HNO_3$  and 150 ml. water and Cool. Gradually add molybdate solution to citric acid-nitric acid mixture with stirring. Dissolve 5 ml. synthetic quinoline in mixture of 35 ml  $HNO_3$  and 100 ml. water. Gradually add this solution to molybdate citric-nitric acid solution mix and let it stand for 24 hours. Filter, add, 28 ml. acetone, dilute to 1 litre with water and mix well. Store in polyethylene bottle.

#### (b) Procedure

(1) Treat 1 gm. sample as prescribed in 4 (i) and dilute it to 200 ml.

(2) Pipette into 500 ml. erlenmeyer flask, aliquot containing not more than 25 mg.  $P_2O_5$  dilute to approximately 100 ml. with water. Proceed with one of the following methods.

A. Add 30 ml. citric molybdic acid reagent and boil gently for 3

minutes. (Solution must be precipitate free at this stage). Remove from heat and swirl carefully. Immediately add from burette 10 ml. quinoline solution with continuous swirling. (Add first 3-4 ml. dropwise and remainder in steady stream) or

B Add 50 ml. quimociac reagent, cover with water glass plate on hot plate in well ventilated hood, and boil for minute.

After treatment with A or B, cool to room temperature, swirl carefully 3-4 times during cooling, filter into gooch with glass fibre filter paper previously dried at 250°C and weighed, and wash five times with 25 ml. portion of water. Dry crucible and contents for 30 minutes at 250°C. Cool in dessicator to constant weight as  $(C_6H_7N)_3H_3PO_4 \cdot 12MoO_3$ . Subtract weight reagent blank. Multiply by 0.03207 to obtain weight of  $P_2O_5$ . Report as per cent  $P_2O_5$ .

(iii) Determination of water soluble phosphorous.

#### (a) Procedure

(1) Place 1 gm. sample on 9 cm. filter paper and wash with small portion of water until filtrate measure approximately 250 ml.

(2) Let each portion pass through filter before adding more and use suction if washing would not otherwise be complete within 1 hour.

(3) If the filtrate is turbid, add 1-2 ml.  $HNO_3$  dilute to 250 ml. and mix.

(4) Pipette into 500 ml. Erlenmeyer flask aliquot containing not more than 25 mg.  $P_2O_5$ .

(5) Dilute if necessary to 50 ml.

(6) Add 10 ml.  $HNO_3$  (1 plus 1) and boil gently for 10 minutes. Cool and dilute to 100 ml. and proceed as 4(ii) (b) (2)(B).

(Reference "Methods of Analysis" A.O.A.C., 1965).

#### (iv) Citrate Insoluble phosphorous.

#### (a) Reagents

(1) Ammonium citrate solution- should have specific gravity of 1.09 at 20°C and pH of 7.0 as determined electrometrically.

Dissolve 370 gm crystalline citric acid in 1.5 litre distilled water and nearly neutralized by adding 345 ml.  $NH_4OH$  (28-

2%  $\text{NH}_3$ ). If concentration of ammonia is less than 28 per cent add correspondingly larger volume, and dissolve citric acid in correspondingly smaller volume of water, Cool and check pH. Adjust with  $\text{NH}_4\text{OH}$  (1 plus 7) or citric acid solution to pH<sub>7</sub>. Dilute solution if necessary to specific gravity of 1.09 at 20°C (Volume will be approximately 2 litres). Keep in tightly stopped bottles and check pH from time to time. If pH has changed from 7.0, readjust.

(2) Other reagents and solutions as in 4 (i) and 4(ii).

(b) Procedure (Acidulated samples and mixed fertilisers).

(1) After removing water soluble  $\text{P}_2\text{O}_5$  in 4(iii) transfer filter and residue within 1 hour to 200 or 250 ml. flask containing 100 ml. ammonium acetate solution previously heated to 65°C.

(2) Close flask tightly with smooth rubber stopper.

(3) Shake vigorously until paper is reduced to pulp and relieve pressure by removing stopper momentarily.

(4) Continuously agitate contents of stopper flask in apparatus equipped to hold contents of flask at exactly 65°C. (Action of apparatus should be such that dispersion of sample in citrate solution is continually maintained and entire inner surface of flask and stopper is continually bathed with solution).

(5) Exactly 1 hour after adding filter and residue, remove flask from apparatus, and immediately filter contents by suction as rapidly as possible through Whatman No.5 paper or equivalent, using buchner or ordinary funnel with platinum or other cone.

(6) Wash with distilled water and at 65°C until volume of filtrate is approximately 350 ml. allowing time for through draining before adding more water.

(7) If material is, one that will yield cloudy filtrate, wash with 2 per cent  $\text{NH}_4\text{NO}_3$  solution.

(8) Determine  $\text{P}_2\text{O}_5$  in citrate insoluble residue by one of the following methods:

A. Dry paper and contents, transfer to crucible, ignite until all organic matter is destroyed and digest with 10-15 ml. HCL until all phosphate dissolve, or

B. Treat wet filter, and contents

as in method prescribed in 4(i)(b)(2) (A)(C)(D) or (E).- Dilute solution to 250 ml. or other suitable volume, mix well filter through dry paper and determine  $\text{P}_2\text{O}_5$  as in 4(ii).

(Reference "Methods of Analysis" A.O.A.C., 1965).

(v) Citrate soluble phosphorus

Subtract sum of water soluble and citrate insoluble  $\text{P}_2\text{O}_5$  from total  $\text{P}_2\text{O}_5$  to obtain citrate soluble  $\text{P}_2\text{O}_5$ .

(Reference "Methods of Analysis" A.O.A.C., 1965).

(vi) Citric acid soluble  $\text{P}_2\text{O}_5$  in fertilisers other than basic slag.

(a) Reagents

(1) Concentrated hydrochloric acid.

(2) Concentrated citric acid,

(3) Calcium carbonate finely ground.

(4) 5 N Sodium hydroxide solution.

(5) Dilute hydrochloric acid-Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre.

(6) Citric molybdic acid solution-stir 54 gm. of molybdic anhydride ( $\text{MoO}_3$ ) with 200 ml. of water, add 11 gm of sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdic anhydride dissolves. Dissolve 60 gm. of citric acid in about 250 to 300 ml. of water and add 140 ml. of concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool and if necessary filter the solution through paper pulp pad. Dilute the solution to 1 litre. If the solution is slightly green or blue in colour add dropwise a dilute (0.5 to 1.0 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in dark.

(7) Quinoline solution - Measure 60ml. of concentrated hydrochloric acid and 300 to 400 ml. of water into 1 litre beaker and warm to 70°-80°C. Pour 50 ml. of quinoline in a thin stream into the dilute acid while stirring. When quinolin has dissolved, cool the solution dilute to 1 litre, and if necessary filter through a paper pulp filter.

(8) Sodium hydroxide solution - 0.5 N

carbonate free.

(9) Indicator solution- Mix three volumes of thymol blue solution and two volumes of phenol phthalein solution prepared as follows:

A Thymol blue solution-Dissolve 250 mg. thymol blue in 5.5ml. of 0.1N sodium hydroxide solution and 125 ml. of industrial methylated spirit. Dilute with water to 250 ml.

B Phenolphthalein solution- Dissolve 250 mg. phenolphthalein in 150ml. of industrial methylated spirit and dilute with water to 250 ml.

(10) Hydrochloric acid -0.5N.

(11) Sodium hydroxide solution -0.1 N

(12) Hydrochloric acid - 0.1 N

(13) Surface active agent - 0.5 per cent solution of sodium dedecyl benzene sulphonate suitable.

(b) Preparation of solution.

(1) Weigh to the nearest mg. about 5 mg. of the sample and transfer to a stoppered bottle of about 1 litre capacity.

(2) Dissolve 10 gm. of pure crystallized citric acid (monohydrate) in water, dilute to 500 ml. and adjust the temperature to 20°C.

(3) Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking.

(4) Shake the bottle continuously for 30 minutes.

(5) Pour the whole of the liquid at once on to a large medium- fine-filter and collect the filtrate, if the filtrate is not clear, pass it again through the same filter.

(c) Procedure

(1) Transfer a volume of the solution prepared according to procedure given above containing less than 70 mg. of phosphoric acid and preferably about 50 mg. to a 500 ml. stoppered conical flask marked at 150 ml.

(2) Dilute the solution with water to 100 ml.

(3) If the sample does not contain calcium add 100 to 200 mg. of calcium carbonate.

(4) Then add 5 N sodium hydroxide

solution dropwise until a faint permanent turbidity or precipitate is formed.

(5) Dissolve the precipitate by the dropwise addition of dilute hydrochloric acid, but avoid an excess.

(6) Dilute to 150 ml. and add 50 ml. of the citric molybdic acid reagent, and heat the solution to incipient abulition, maintain it at this temperature for 3 minutes and then bring it to the boiling point.

(7) From burette slowly add 25 ml. of the quinoline solution with constant swirling throughout the first few ml. being added dropwise, the rest in slow stream.

(8) Keep the solution gently boiling during the addition.

(9) Immerse the flask in boiling water for 5 minutes, then cool it to 15°C, in running water.

(10) Filter with suction the contents of the flask on a paper pulp pad and wash the flask precipitate and filter with successive small washes of cold water until they are free from acid.

(11) Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask.

(12) If necessary, wipe the funnel with small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask.

(13) Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

(14) Remove the stopper and wash it with a water, returning the washing of the flask.

(15) Add a measured volume of 0.5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml. in excess.

(16) Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after the addition of 0.5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary).

(17) Add 0.5 - 1.0 ml. of the indicator solution, and titrate the excess of sodium hydroxide with 0.5 N hydrochloric acid, until the indicator changes from violet to green-blue, and then very sharply

to yellow at the end point.

(18) Deduct the number of ml of 0.5 N hydrochloric acid used from the number of ml. 0.5 N sodium hydroxide, to ascertain the volume of 0.5 N sodium hydroxide equivalent to the phosphoric acid.

(19) Carry out a blank determination on all the reagents, omitting only the sample, and using 0.1 N standard alkali and acid instead of 0.5 N for the titration. Calculate the blank in terms of 0.5 N alkali and subtract it from the original result.

(20) Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1.0 ml. of 0.5 N sodium hydroxide = 1.366 mg.  $P_2O_5$ .

(Reference Statutory Instruments 1960) No. 1165, Agriculture, the fertiliser and feeding stuffs Regulations, 1960).

(vii) Free phosphoric acid as  $(P_2O_5)$

(a) Reagents

- (1) Acetone-conforming to IS: 170-1950 specification for acetone.
- (2) Standard sodium hydroxide solution-0.1 N.
- (3) Bromocresol green indicator solution-dissolve 0.1 gm of bromicresol green in 100 ml. of rectified spirit conforming to IS:323-1959 specification for rectified spirit (Revised).

(b) Procedure

- (1) Weigh accurately about 2.5 mg. of the prepared sample and transfer to a soxhlet extract or
- (2) Add about 100 ml. of acetone and extract for three hours.
- (3) Cool and distil off the acetone as far as possible.
- (4) Take up the residue with water and make up the volume to 250 ml.
- (5) Pipette out exactly 100 ml. of this solution and titrate with standard sodium hydroxide solution, using bromocresol green as indicator until colour just changes from yellow to blue.

Calculations

Free phosphoric acid (as  $P_2O_5$ ) per cent by weight

$$= \frac{17.75 - XV - XN}{W}$$

Where

V = volume in ml. of standard sodium hydroxide solution used.

N = normality of standard sodium hydroxide solution, and

W = weight in gm. of sample taken for the test.

(Reference-IS Specification for superphosphate)

(Revised)

(IS. 294 - 1962)

5. Determination of potassium

Determination of potassium in all kinds of fertilisers is given in this section. Two alternative methods have been described. Any of these two methods may be used depending upon the availability of reagent, and suitability of the method.

(i) Perchloric acid method

(This method depends on the insolubility of potassium perchlorate and the solubility of sodium perchlorate in alcohol and is applicable in presence of alkali metals chlorides and nitrates. Sulphates and ammonium salts must be absent on account of the low solubility of sodium sulphate of ammonium perchlorate in alcohol. Phosphates must be removed. Methods are given for the elimination of the effect of interfering substances).

(a) Reagents

- (1) Concentrated hydrochloric acid.
- (2) Barium chloride solution. Dissolve 100 gm. of barium chloride in water, filter the solution and dilute to 1 litre.
- (3) Dilute hydrochloric acid- Dilute 240 ml. of concentrated hydrochloric acid with water to one litre.
- (4) Calcium oxide - finely ground.
- (5) Ammonium hydroxide solution - sp. gr. 0.88.
- (6) Ammonium carbonate solution - saturated aqueous solution.
- (7) Ammonium oxalate solution- saturated aqueous solution.
- (8) 20 per cent perchloric acid solution.

- (9) Alcohol - industrial methylated spirit 95-96 per cent V/V.
- (10) Wash solution-Add potassium perchlorate to alcohol and shake until saturated solution is obtained. Keep the solution over solid potassium perchlorate and filter immediately before use.
- (b) Potassium salts free from sulphates and other interfering substances.
- (1) Dissolve in water a portion of the sample weighed to the nearest mg equivalent to potassium content to 1.5 to 2.0 gm of potash.
  - (2) Cool the solution to 20°C, dilute to 500 ml in a volumetric flask, mix well and filter through a dry filter.
  - (3) Determine the potash in 50 ml of solution by precipitating with perchloric acid as described under procedure.
- (c) Potassium salts with sulphates or other interfering substances (The following method is given for eliminating the interference caused by presence of sulphate. If salts contain phosphates, iron, manganese or substances other than sulphate that interfere with the determination of potash, the method described for mixed fertiliser should be used).
- (1) Weigh to the nearest mg a portion of the sample equivalent in potassium content to 1.5 to 2.0 gm of potash, into a 50 ml beaker. Add about 300 ml of water and 20 ml concentrated hydrochloric acid and heat the solution to boiling.
  - (2) To the boiling solution cautiously add, drop by drop barium chloride solution in an amount slightly in excess of that previously determined as necessary to ensure the complete precipitation of sulphate.
  - (3) Cool the liquid to 20°C, transfer to a 500 ml volumetric flask, dilute to 500 ml, mix and filter through a dry filter.
  - (4) Take 50 ml of the filtrate and evaporate to dryness in a basin, moisten the residue with concentrated hydrochloric acid.
  - (5) Again evaporate to dryness, dissolve the residue with 5-10 ml dilute hydrochloric acid and filter, if necessary. Determine the potash in solution by the method described under procedure.
- (d) Potassium in mixed fertilizers.
- (1) Weigh to nearest centigram about 10 gm of the sample and if organic matter is present, gently incinerate at temperature not exceeding 500°C.
  - (2) Transfer the weighed portion of the sample or the incinerated residue to a 500 ml breaker with a little water, add 10 ml concentrated hydrochloric acid and then warm for ten minutes.
  - (3) Dilute with water to about 300ml and bring gradually to boiling point.
  - (4) Add 10 gm of calcium oxide made into a paste with water.
  - (5) Bring contents again gently to the boiling point, and keep so heated for about half an hour with frequent stirring.
  - (6) Cool to 20°C, transfer to a 500ml volumetric flask, dilute to 500 ml, and after thoroughly shaking filter through a dry filter paper.
  - (7) Transfer 250 ml of the filtrate to another 500 ml volumetric flask, make just acid with hydrochloric acid and heat to boiling point.
  - (8) To the boiling solution cautiously and drop by drop, barium chloride solution until there is no further precipitation of barium sulphate.
  - (9) Render the contents of the flask alkaline with ammonium hydroxide solution and precipitate the calcium and any excess of barium by adding ammonium carbonate solution until no further visible precipitation occurs followed by the addition of about 1 ml of ammonium oxalate solution.
  - (10) Cool to 20°C, dilute with water to 500 ml and after thoroughly shaking, filter through a dry filter paper.
  - (11) Measure 100 ml of the filtrate and evaporate to dryness in a basin. Expel the ammonium salts from the residue by gently heating

the basin over a low flame, being careful to keep the temperature below that of faint redness.

- (12) Cool the residue, moisten with concentrated hydrochloric acid and again evaporate to dryness.
- (13) Take up the residue with water and filter if necessary.
- (14) Determine the potash in the solution by precipitation with perchloric acids described under procedure.

(e) Procedure

- (1) Transfer the solution obtained as described above into a basin and add about 7 ml of perchloric acid solution.
- (2) Place the basin on a hot plate or sand bath and evaporate the contents until the white fumes are copiously evolved.
- (3) Cool and dissolve the precipitate in a little hot water.
- (4) Add about 1 ml of perchloric acid solution and again concentrate to the fuming stage.
- (5) Thoroughly cool the residue in the basin and stir in 20 ml of alcohol.
- (6) Allow the precipitate to cool and settle, then pour the clear liquid through a dry filter paper, draining the precipitate in the basin and stir in 20 ml of alcohol.
- (7) Allow the precipitate to cool and settle, then pour the clear liquid through a dry filter paper, drain the precipitate in the basin as completely as possible.
- (8) Redissolve the precipitate on the paper and that remaining in the basin with hot water.
- (9) Add 2 ml of perchloric acid solution to the combined solution and evaporate the whole down to the fuming stage.
- (10) Cool the residue in the basin and thoroughly stir the contents with 20 ml of alcohol.
- (11) Allow the precipitate to cool and settle and pour the clear liquid through a weighed gooch or sintered

glass crucible, draining the precipitate as completely as possible from the liquid before adding 5 ml of wash solution.

- (12) Wash the precipitate by decantation with several similar small portions of the wash solution, pouring the washing through the crucible.
- (13) Transfer the precipitate to the crucible and wash it well with the wash solution until free from acid.
- (14) Dry the precipitate at 100°C and weigh.
- (15) Regard the precipitate as potassium perchlorate ( $KClO_4$ ), and calculate its equivalent as potash ( $K_2O$ ) by multiplying its weight by 0.34.

(Reference- Statutory Instruments 1960, No. 1165, Agriculture, The Fertiliser and Feeding Stuffs Regulation, 1960).

(ii) Sodium tetraphenylboron method.

This method is applicable to both mixed and straight potassium fertilisers.

Reagents.

- (1) Sodium hydroxide solution - 20 per cent. Dissolve 20 gm  $NaOH$  in 100 ml distilled water.
- (2) Formaldehyde solution - 37 per cent
- (3) Sodium tetraphenyl boron (STPB) solution approximately 1.2 per cent.

Dissolve 12 gm sodium tetraphenyl boron in approximately 800 ml water. Add 20-25 ml  $Al(OH)_3$ , stir for 5 minutes, and filter (Whatman No. 42 paper or equivalent) into 1 litre volumetric flask. Rinse breaker sparingly with water and add to filter. Collect entire filtrate, add 2 ml 20 per cent  $NaOH$  solution, dilute to volume with water, and mix. Let it stand for 48 hours and standardize. Adjust to that 1 ml STPB-1 per cent  $K_2O$ . Store at room temperature.

- (4) Quaternary ammonium chloride solution approximately 0.625 per cent.

Dilute 50 ml of 12.8 per cent Zephiran chloride to 1 litre with water, mix and standardize. Cetyltrimethyl ammonium bromide may be substituted for Zephiran chloride. If other concentration is used, adjust volume.

- (5) Clayton yellow - 0.04 per cent. Dissolve

40 mg in 100 ml water.

(b) Standardization of solutions.

- (1) Zephiran chloride - To 1.0 ml STPB solution in 125 ml. Erlenmeyer flask, add 20-25 ml water. 1 ml 20 per cent  $\text{NaOH}$  2.5 ml  $\text{HCHO}$ , 1.5 ml 4 per cent  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  and 6-8 drops of indicator (5) above.
- (2) Titrate to pink end point with Zephiran chloride solution, using 10 ml semimicro burette. Adjust Zephiran chloride solution so that 2.0 ml = 1.0 ml STPB solution.
- (3) Sodium tetraphenyl boron solution- Dissolve 2.5 gm of  $\text{KH}_2\text{PO}_4$  in water in 250 ml volumetric flask, add 50 ml 4 per cent  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  solution, dilute to volume with water and mix. (It is not necessary to bring to boil). Transfer 15 ml aliquot (51.92 mg  $\text{K}_2\text{O}$  43.10 mg K) to 100 ml volumetric flask, add 2 ml 20 per cent  $\text{NaOH}$ , 5 ml  $\text{HCHO}$  and 43 ml STPB reagent. Dilute volume with water, mix thoroughly, let stand 5-10 minutes and pass through dry filter. Transfer 50 ml aliquot of filtrate to 125 ml Erlenmeyer flask, add 6-8 drops of indicator (5) above and titrate excess reagent with Zephiran solution. Calculate titration values as follows:

$F = 34.61 / (43 \text{ ml-ml Zephiran})$   
 $= \% \text{K}_2\text{O/ml STPB reagent}$ . Factor applies to all fertilizers if 2.5 gm sample is diluted to 250 ml and 15 ml aliquot is taken for analysis. If results are to be expressed as K rather than  $\text{K}_2\text{O}$ , substitute 28.73 for 34.61 in calculating the value of F.

(c) Preparation of solution.

(1) Mixed fertiliser

- A. Place 2.5 gm sample or factor weight 2.430 gm in 250 ml volumetric flask.
- B. Add 125 ml water and 50 ml saturated  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  solution. Add 1 ml of diglycol stearate solution if needed to prevent foaming.
- C. Boil for 30 minutes, add slight excess of  $\text{NH}_4\text{OH}$  and after cooling

dilute to 250 ml. Mix and pass through dry filter.

- (2) Potassium salts (potassium chloride sulphate, potassium magnesium sulphate and Kainite)
  - A. Dissolve 2.5 gm or factor weight 2.430 gm and dilute to  $C \times 250$  ml without adding  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ .
  - B. When interfering substances such as  $\text{NH}_4\text{C}_2\text{O}_4$ , Al. etc. are presently proceed as in (1) above.

(d) Procedure

- (1) Transfer 15 ml aliquot of sample solution to 100 ml volumetric flask and add 2 ml 20 per cent  $\text{NaOH}$  and 5 ml  $\text{HCHO}$ .
- (2) Add 1 ml standard STPB solution for each 1 per cent  $\text{K}_2\text{O}$  expected in sample plus additional 8 ml excess to ensure complete precipitation.
- (3) Dilute to volume with water, mix thoroughly, let it stand for 5-10 minutes and pass it through dry filter (Whatman 12 or equivalent).
- (4) Transfer 50 ml filtrate to 125 ml Erlenmeyer flask, add 6-8 drops of indicator (5) and titrate excess reagent with standard Zephiran solution. Per cent  $\text{K}_2\text{O}$  in the sample = (ml STPB added = ml Zephiran)  $\times F$  where  $F$  = per cent- $\text{K}_2\text{O/ml/STPB}$  = reagent.

(Reference-"methods of Analysis", A.O.A.C. 1965).

6. Method of Analysis of Anhydrous Ammonia

(i) Estimation of Water and Ammonia.

(a) Scope

This method is for the determination of water at  $20^\circ\text{C}$  (or room temperature), and usually ammonia by difference) of any essentially anhydrous ammonia liquid product. Normally, the water will be low (less than 0.5 per cent) or sample of synthetic ammonia.

(b) Apparatus

- (1) Ammonia Sampling tubes.
- (2) Fume hood with exhaust dust.
- (3) Sample tube holder rack in hood fabricated as convenient to hold number of tubes desired.



(4) Gloves, protective, elbow length.

(c) Reagents

Charcoal, reagent, 14-20 mesh.

(d) Procedure

(1) Remove the sample tubes from the sample carrier and place in the provided sample tube holders in the fume hood.

(2) Allow the ammonia sample to boil and to evaporate (approximately 30 minutes) spontaneously in the fume hood. Because of the low temperature of the boiling ammonia, atmospheric moisture freezes on the sample container. Exercise care to see that none of this ice contaminates the sample.

(3) When the boiling of the sample ceases, remove the tube from the holder. Wipe the outer surface of the tube dry and read and record the volume of liquid residue in the tube.

(4) From the volume recorded, calculate the weight per cent water in the sample. Ignore the small piece of charcoal, if used. Calculation

$$\text{percent water} = \frac{A \times F \times 0.890 \times 0.684}{V \times 0.682} \times 100$$

Where A = Volume of residue in tube (ml)

V = Volume of sample taken (ml)

F = Evaporation factor, taken from Table 1 corresponding to noted sample pressure when sample was obtained.

0.890 = density of residue (gm/ml).

0.684 = weight fraction of water in residue.

0.682 = density of sample (gm/ml).

(e) Precaution.

The evaporation in the fume hood should be conducted with the window lowered to protect personnel from possible spray. Whenever it is necessary to handle the sample tubes, protective gloves should be worn.

Note: Determination of residue on evaporation will not usually be necessary for normal ammonia sample. If sample is clear and water residue is clear and colourless, the residue may be considered to be nil. However, should it become necessary to determine the dissolved solids and suspended solids in anhydrous ammonia, proceed as follows:

(f) Sampling

Thoroughly clean and dry the sample tubes, weigh ( $W_1$ ) each selected tube to the nearest milligram with a similar tube as counterpoise. Obtain the samples as desired earlier.

(g) Apparatus.

(1) Analytical balance.

(2) Ammonia sampling tubes.

(3) Rubber tubing, 1/4" OD length desired.

(h) Reagents

Air dry, carbon dioxide -free.

(i) Procedure.

(1) Start test following elimination of the water content of the sample as given earlier.

(2) Connect one end of a rubber hose to a source of dry, carbon dioxide-free air. Insert the other end of the hose into the sample tube and gently force air through the sample tube until all liquid has been evaporated and no ammonia gas remains in the tube.

(3) Wipe a counterpoise and the sample tube containing the residue to substantially the same extent to remove moisture which may have condensed on their outer surfaces.

(4) weigh the sample tube and residue to nearest milligram.

(5) Calculate the weight per cent residue of the sample.

Calculation

$$\text{per cent residue} = \frac{(W_2 - W_1) \times F \times 100}{V \times 0.682}$$

Where  $W_1$  = weight (gm) of tube (before sampling).

$W_2$  = weight (gm) of tube and residue.

V = Volume of sample taken (ml)

F = Evaporation factor, taken from Table 1 corresponding to noted sample pressure when sample was obtained.

0.682 = density of sample (gm/ml).

(j) Precaution

A check of completeness of evaporation

may be made by visual inspection and by carefully smelling the air in the tube making sure that no carbon dioxide or moisture from the breath enters the sample tube.

(k) Percent ammonia content (by difference)  
In general, the ammonia content of the sample will be : per cent ammonia = 100-(per cent water- per cent residue, if determined).

(1) Precaution

The evaporation in the fume hood should be conducted with the window lowered to protect personnel from possible spray. Whenever it is necessary to handle the sample tubes, protective gloves should be worn.

(ii) Determination of oil content.

(a) Apparatus.

(1) 500 ml conical flask calibrated to hold 300 ml (about 200 gms) of sample and with a bunk fitted with glass through which the exist gas can be led away to a safe place. Ensure that the glass tube and exist line are free from constructions.

(2) Platinum dish 75 mm in diameter.

(b) Reagent.

The reagent used shall be of a recognised analytical reagent quality like petroleum spirit with a boiling range of 40° to 60°C.

(c) Procedure

(1) Quickly running the sample (as per the sample procedure) upto the calibration mark, insert the bunk.

(2) Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly.

(3) When the evaporation is complete, remove the bunk and gently blow out the last traces of ammonia with a small jet of filtered air free from carbon dioxide.

(4) Dry the outside of the flask.

(5) Place the flask in an oven at 105±5°C for 15 minutes to remove the moisture blowing out the last traces with a gently stream of filtered air at the end of this period; allow to cool.

(6) Add to the flask approximately 100 ml of the petroleum spirit and swirl to dissolve the oil.

(7) Filter the solution through a

small filter paper (a Whatman No.31 paper is suitable) direct into the platinum dish previously traced to the nearest 0.1 mg.

(8) Repeat the operation with two more successive 10 ml portions of petroleum spirit, filtering through the same paper as before into the same dish.

(9) Evaporate the combined extracts to dryness on a water bath in a fume cup board, taking care to avoid naked lights and finally dry in an oven 105-5°C for 30 minutes.

(10) Allow the dish to cool thorough in a desicator and weigh again to the nearest 0.1 mg.

(11) At the same time, carry out a blank determination on the petroleum spirit and filter paper.

(12) Make an appropriate correction in the calculation.

Calculation

oil content, parts per million by weight=

$$\frac{(W_1 - W_2) - W_3}{0.68 V_2} \times 10^6 F$$

Where  $W_1$  = weight in grams of platinum dish and residue.

$W_2$  = weight in grams of dish alone.

$W_3$  = weight in grams of oil in blank determination.

$V_2$  = volume in ml of sample taken.

F = evaporation factor taken from Table 1 corresponding to noted sample pressure when sample was obtained.

Table 1 - Evaporation factor for different vessels or line pressures.

Vessel or Line Pressure (Psig)	Evaporation Factor (F)
1	2
0	1.000
10	0.963
20	0.940
30	0.920
40	0.900
50	0.885
60	0.870
70	0.860
80	0.850
90	0.840
100	0.830
110	0.821
120	0.813

130	0.805
140	0.797
150	0.789
160	0.782
170	0.776
180	0.770
190	0.764
200	0.758

Note: When a sample of liquid ammonia is transferred to an open flask/tube from a container in which the pressure is higher than the atmospheric that sample quickly reaches the thermodynamics equilibrium with its new environment. Some of the liquid ammonia will evaporate and since the ammonia so lost as vapour contains non-volatile constituents present in the original product, the concentration of these constituents will increase in the liquid sample taken. This may be significant factor is subsequent analysis and fortunately it is possible to calculate from the known thermodynamic properties of ammonia an appropriate correction. This correction is usually known in this context at the evaporation factor of flask factor and is simply that fraction by weight of the original liquid ammonia which remains as liquid in the sample. Multiplication of the determined quantity of a non-volatile constituents (oil, water dissolved or suspended solids, etc.) by the evaporation factor gives a result which will be closer to the true figure. The Table above presents the evaporation factor for different vessel or line pressure.

When a sample is transferred (example by gravity) from a container of a cylinder or the sampler at the same pressure, there should be no loss by evaporation and in this case it is not necessary to use evaporation factor.

#### 7. Method of analysis of Zinc Sulphate, Agricultural Grade.

##### (i) Quality of Reagents.-

Pure chemicals and distilled water shall be used in tests.

Note: 'Pure chemicals' shall mean chemicals that do not contain impurities which effect the results of analysis.

##### (ii) Determination of Matter Insoluble in Water.-

###### (a) Reagents

Dilute Sulphuric Acid - 10 per cent

###### (b) Procedure

Dissolve 25.0 g of the material in 125 ml of water and add 1 ml of dilute sulphuric acid. Heat the solution to boiling, filter through a weighed and prepared Gooch crucible or sintered glass crucible (G.No.4) and wash the residue thoroughly with hot water. Dry the crucible at  $110^{\circ} \pm 5^{\circ}$  to constant mass.

###### Calculation

Matter insoluble in water,

per cent by mass weight = 4A

Where A = weight in g of the residue

###### (iii) Determination of Zinc.-

###### (a) Reagents

(1) Disodium Ethylenediamine Tetra Acetate (EDTA) Solution-Dissolve 18.6 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to 1 litre in a volumetric flask.

(2) Standard Zinc Solution - weigh 1.25 gm of reagent grade zinc metal, dissolve in 20 ml of hydrochloric acid (1 : 1) and make up to 500 ml in a volumetric flask.

(3) Ammonium hydroxide - 20 per cent (m/m).

(4) Eriochrome Black -T indicator-Mix thoroughly 1 g of eriochrome black-T with 100 g of sodium chloride.

(5) Ammonium Chloride.

(6) \* Sodium Cyanide.

(7) Formaldehyde Solution - Dilute 1 volume of formaldehyde 36 per cent (m/v) with 8 volumes of water.

###### (b) Procedure

(1) Standardisation of EDTA solution Pipette 50 ml of standard zinc solution into 600 ml beaker. Add ammonium hydroxide until a permanent precipitate is formed, followed by 10 ml more of the alkali. Dilute to 500 ml and add a few specks of the indicator. Titrate the red solution with EDTA solution to a clear blue end point.

(2) Dissolve 0.5 g of the material, accurately weighed in about 25 ml of water and add 0.1 g of ammonium chloride and 0.5 g of sodium cyanide. Add more

\* To be used with extreme care

cyanide if the precipitate formed does not dissolve completely. Add approximately 75 ml of water and 10 to 20 ml of ammonium hydroxide. Add sufficient amount of ariochrome black T indicator to produce a red colour (only a few specks are required), and stir until dissolved. Add 20 ml of formaldehyde solution and titrate with EDTA solution to a clear blue end point. (it is important to carry out the titration immediately after addition of formaldehyde solution).

#### Calculation

Zinc (as Zn), per cent by mass =

$$12.5 \frac{V_1}{V_2 M}$$

where

$V_1$  = Volume in ml of EDTA solution used with the material in (2)

$V_2$  = Volume in ml. of EDTA solution used with standard zinc solution in (1) and

M = mass in g of the material taken for the test.

#### (iv) Colorimetric Method for Determining of Copper.

(a) Outline of the method - Sodium diethyldithio carbonate reacts with slightly acidic or ammoniacal solution of copper in low concentration to produce a brown colloidal suspension of the cupric diethyldithiocarbamate. The suspension can be extracted with an organic solvent and the colour extracted determined spectrophotometrically.

#### (b) Apparatus

Photometer - Any suitable photoelectric colorimeter.

#### (c) Reagents

(1) Ammonium Citrate Solution - 40 per cent (m/v).

(2) Sodium Diethyldithiocarbamate Solution - 0.1 per cent (m/v).

(3) Standard Copper Solution - Clean the surface of a copper wire with sand paper. Dissolve 100 mg of pure copper in dilute nitric acid. Heat the solution nearly to dryness to drive off the acid. Add about 10 ml of water and heat again nearly to dryness. Take up the residue in water, add 25 ml of 1N sulphuric acid and dilute to 1,000 ml. One millilitre of this solution contains 0.1 mg of copper (as Cu).

Alternatively, dissolve 3.928 g of cupric sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in sufficient water containing 1 or 2 ml of concentrated sulphuric acid and dilute to 1000 ml. One millilitre of the solution contains 1.0 mg of copper (as Cu).

(4) Ammonium Hydroxide - 18 N.

(5) Carbon Tetrachloride.

(d) Preparation of sample.

(1) Weigh 5 g of sample accurately, dissolve in distilled water and add 1 ml. of sulphuric acid. Filter the solution and make up to 250 ml with water in volumetric flask.

Take 50 ml. of the above solution in a beaker, heat, pass hydrogen sulphide gas or add sodium sulphide solution and ensure complete precipitation. Filter hot and keep the filtrate for subsequent analysis.

(2) Boil the residue with dilute nitric acid and filter, if necessary. To the filtered solution add sulphuric acid, evaporate, dilute and filter. Keep the residue for determination of lead.

(e) Procedure for determination of copper

Determine copper in the filtrate from (d) (2) above by diethyldithiocarbamate method as detailed below:-

Transfer an aliquot containing not more than 0.55 mg of copper to a 100 ml. separating funnel. Add 2 ml of ammonium citrate solution and adjust the pH to 8.5 with ammonium hydroxide. The presence of ammonium ions helps to dionize iron, if present, and to prevent rapid fading of developed colour. Add 10 ml. of sodium diethyldithiocarbamate solution and develop the colour. Extract the coloured complex with 5-10 ml. of carbon tetrachloride. Separate the carbon tetrachloride layer from aqueous layer and centrifuge for 5 minutes to separate the water droplets. Transfer the carbon tetrachloride solution to a photometric cell and measure its absorbance at the wavelength of maximum absorption 440 ml relative to reagent blank. Calculate the corrected absorbance by subtracting the reading obtained for the solution containing no copper.

Transfer to a series of 100 ml separating funnels aliquots of standard copper solution corresponding to 0, 0.1, 0.2, 0.5, 1.0, 2.0, 2.5 mg of copper and proceed exactly as prescribed above. Plot a graph of corrected, absorbance of solutions against their copper contents.

Note : If bismuth is suspected it has to be separated or otherwise accounted for.

#### Calculation

Calculate the corrected absorbance by subtracting the value obtained for the blank from that obtained for the test solution and read from the calibration curve the corresponding mass of copper.

Copper (as Cu), per cent by mass =

$$\frac{M_1 \times 100}{M_2}$$

Where

$M_1$  = Mass in g of copper as determined in the given aliquot of the test solution, and

$M_2$  = mass in g of the material present in the aliquot of the test solution

(v) Determination of lead by colorimetric method using Dithizone.-

#### (a) Apparatus

Nessler Cylinders - 50 ml. capacity.

#### (b) Reagents

(1) Standard Lead Solution - Dissolve 0.40 g of lead nitrate [ $Pb(NO_3)_2$ ] in water containing 2 or 3 ml. of concentrated nitric acid and make up the volume to 1000 ml. with water. Transfer 10 ml. of this solution to volumetric flask, add 2 or 3 ml. of concentrated nitric acid and dilute with water to 1000 ml. One millilitre of this solution contains 2.5 mg of lead (as Pb). The diluted solution shall be freshly prepared.

(2) Dilute Ammonium Hydroxide - 1N, approximately.

(3) Reagent A - Dissolve 25 g of triammonium citrate or 22 g of citric acid and 4 g of hydroxylamine hydrochloride in about 200 ml of water. Add dilute ammonium hydroxide to bring the pH to 8.5. Dilute the solution to 500 ml. Purify this solution by extracting with 15 ml portions of 0.01 per cent dithizone solution until the final colour of the dithizone extract is green. Wash the aqueous remainder portion three times with 25 ml portions of chloroform and finally with 25 ml portion of carbon tetrachloride.

(4) Dithizone (Diphenyl thicarbazon or phenylazothimoformic Acid) Solution - Dissolve 0.01 g of dithizone in 100 ml of carbon tetrachloride, shaking

intermittently for 1 hour. Allow to stand overnight and shake once again before using. This shall be kept in a cool and dark place. This gives a 0.01 per cent solution. Filter, if necessary. Dilute 10 ml of this solution to 100 ml with carbon tetrachloride in a 100 ml volumetric flask. This shall be prepared fresh before determination. This gives a solution of 0.001 per cent.

Note: 1. Carbon tetrachloride used should be further purified. One litre of carbon tetrachloride is extracted with two portions of 25 ml dilute ammonium hydroxide. and then kept over 100 g of activated carbon. Before use, it is decanted and distilled at about 80°C over a little fresh lime.

Note 2. Sometimes dithizone solid and its 0.01 per cent solution deteriorate on storage. The 0.01 per cent solution should, therefore, be tested before further dilution, by shaking 2 ml of the solution with 5 ml of 1 per cent ammonium hydroxide. If the organic layer is only faintly yellow under these conditions, the solution may be used. If it is deeply coloured it shall be discarded and fresh solution be prepared. The solution as well as the reagent should be stored in a refrigerator and exposure to sunlight should be avoided during analytical work. To increase the stability of 0.01 per cent solution, it should be covered with a thin aqueous layer saturated with sulphur dioxide.

(5) Thymol Blue indicator Solution- 1 per cent (m/v) solution in rectified spirit.

#### (c) Procedure

(1) Take several aliquots of standard lead solution into a series of separating funnels, add 5 ml of the water and 10 ml of the Reagent A. Then add 2 drops thymol blue indicator and bring the pH of the solution to 8.5 by addition of dilute ammonium hydroxide. Add 5 ml of the dithizone solution and shake well for about 10 seconds. Drain the organic layers into stoppered Nessler cylinders.

(2) Dissolve the residue obtained in iv(d)(2) above. in dilute nitric acid

and dilute with water. Take a suitable aliquot of the solution and transfer to a separating funnel. Develop the colour as prescribed above. Drain the organic layer into a stoppered Nessler cylinder, add 10 ml of dilute ammonium hydroxide, and shake for about 10 seconds. Compare the colour developed with those of the standard solutions. Note the volume of the standard with which the colour of the test solution matches.

Note : If the colour of the test solution is intermediate between two standard solutions, then the expenditure is repeated by taking more number of standard solutions in that range and exact colour matching is arrived at.

#### Calculation:

$$\text{Heavy metals(as Pb), per cent by mass} = \frac{100 \times V \times f}{M}$$

where

V = Volume in ml of standard lead solution matching with the test solution.

f = mass in g of heavy metals (as Pb) equivalent to 1 ml of standard lead solution, and

M = mass in g of the material in the aliquot taken for the test.

(vi) Determination of Magnesium in the Sample material.-

#### (a) Reagents

(1) Eriochrome Black T Indicators- Dissolve 0.1 g of eriochrome black T in 25 ml of methyl alcohol.

(2) Ammonium Hydroxide - Ammonium Chloride Buffer Solution - Mix. 350 ml of ammonium hydroxide (20 per cent w/w) with 54 g of ammonium chloride. Dilute with water and make up the volume to 1000 ml (The pH of the solution should not be more than 10).

(3) Standard Magnesium Solution.- 0.01 M Weigh 2.4640 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) and dissolve it in water. Make up the volume to one litre.

(4) Ethylenediamine Tetra-Acetate (EDTA) Solution-Dissolve 3.72 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to one litre.

#### (b) Procedure

Standardization of EDTA Solution-Take 10 ml of standard magnesium solution in a conical flask. Add 20 ml of water,

one millilitre of eriochrome black T indicator and 25 ml of ammonium hydroxide ammonium chloride buffer solution. Heat at 40°C to 50°C and then titrate with EDTA solution, maintaining the temperature between 40°C and 50°C until the colour changes from wine red to distinct blue.

$$\text{Molarity of EDTA solution} = \frac{10M_1}{V_1}$$

where

$M_1$  = molarity of standard magnesium solution, and

$V_1$  = Volume in ml of EDTA solution used for titration.

(c) Determination of Magnesium in the sample material-Take the filtrate from (iv) (d) (1) (after removal of copper) add a few drops of concentrated nitric acid, boil and cool and then add solid ammonium chloride (about 2 grams), boil and cool, add ammonium-hydroxide till the strong smell of ammonia comes and filter the precipitate through sintered crucible, take this filtrate and add dilute sulphuric acid till the solution is acidic (test with methyl red), heat the solution to boil and add excess of di-ammonium-hydrogen phosphate with continuous stirring. Add 10 per cent ammonia solution with continuous stirring till the solution is just alkaline (test with methyl red) white precipitate of zinc ammonium phosphate will be formed the optimum PH for precipitation is 6-7, allow it to stand for 3-4 hours. till the solution is just alkaline (test with methyl red) while precipitate of zinc ammonium phosphate will be formed the optimum pH for precipitation is 6-7, allow it to stand for 3-4 hours. Filter through Whatman filter paper no.40. Collect the filtrate in a volumetric flask. Make up the volume (say 100 ml). Take a suitable Aliquote (say 10 ml) for the determination of magnesium. Add 20 ml of water, one ml of eriochrome black-T indicator and 20 ml of ammonia hydroxide-ammonia chloride buffer solution. Heat to 40°C to 50°C and tirate with standard EDTA solution, maintaining the temperature between 40°C and 50°C until the colour changes from wine red to distinct blue.

$$1 \text{ ml of } 0.01\text{M-EDTA} = 0.2432 \text{ mg of 'Mg'}$$

$$\text{Mg} = \frac{X \times 0.2432}{5}$$

where

X = volume of 0.1M-EDTA used for titration.

The calculation factor 5 is derived presuming that 5 g of material is taken for test and the filtrate obtained is 100

ml out of which 10 ml is titrate.

(vii) Determination of pH.-

Dissolve 5 gm of the material in freshly boiled and cooled water. Dilute to 25 ml and mix. Determine the pH value of the solution with pH meter."

8. Alternate method of analysis of zinc-sulphate, agriculture grade

(i) Quality of Reagents.-

1. Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests.

Note - 'Pure chemicals' means chemicals that do not contain impurities which effect the results of analysis.

'Demineralised water' means the water obtained after passing distilled water through a cation and an anion exchange resins or a combined cation-anion exchange resin.

(ii) Determination of Zinc.-

(a) Reagents

(1) Standard Zinc solution.- Weigh 0.4398 g of zinc sulphate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ -A.R. grade) on a clear watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add one ml of 10 per cent sulphuric acid (A.R. Grade) and make the volume upto the mark. Stopper the flask and shake the solution well. This is 100 ppm Zinc solution hereinafter called Standard A. This solution should be stored in a clean bottle for further use. Dilute 10 ml of 100 ppm solution of Zinc (Standard A) to 100 ml to get 10 ppm standard zinc solution designated as Standard B.

(2) Glass distilled or mineralised water of PH  $2.5 \pm 0.5$ .- Dilute 1 ml of 10 per cent sulphuric acid to one litre with glass distilled or mineralised water and adjust the pH to 2.5 with a pH meter using  $\text{H}_2\text{SO}_4$  or NaOH. This solution is called acidified water and 5 to 10 litres of this solution should be prepared at a time.

(3) Preparation of working standards.- Pipette the following volume of Standard B in 50 ml numbered volumetric flasks and make the volume with acidified water.

Flask No.	Volume of Standard B taken(ml)	Concentration of zinc after making volume to 50 ml(ppm)
1	0.0	0.0
2	1.0	0.2
3	2.0	0.4
4	3.0	0.6
5	4.0	0.8
6	5.0	1.0
7	7.0	1.4
8	9.0	1.8
9	10.0	2.0

Stopper the flasks and shake them well. Prepare the standard in duplicate. The same acidified water should be used for preparing the solution of unknown fertilizer samples. Fresh standards should be prepared every time when a fresh lot of acidified water is prepared.

(b) Procedure

(1) Preparation of Zinc Sulphate fertilizer samples.- Weigh 0.25 g of the material on a clean watch glass and transfer it to one litre volumetric flask through the funnel giving repeated washings with glass distilled water and dissolve the material by shaking well. Then make the volume upto mark with glass distilled water and shake well.

(2) Take 5 ml of the prepared solution in 250 ml volumetric flask and make the volume with acidified water. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flasks. The flasks should be rinsed with a 10 to 15 ml of the filtrate and then continue filtration.

(3) Flaming the solutions.- Flame the standards and the filtered samples on atomic absorption spectrophotometer at a wavelength of 213.8 mu (Zn line of the instrument).

Calculations

Prepare a standard curve of known concentrations of zinc solution by plotting the absorbance values on Y-axis against their respective zinc concentration on X-axis. Calculate the percentage zinc in zinc fertiliser by multiplying zinc concentration value calculated from standard curve by 20.

Example :

Weight of the fertiliser sample = 0.25g  
Volume made = 1000

Further dilution = 50 times  
Reading of the samples from Atomic Absorption = Y  
Corresponding concentration value of zinc from standard curve against Y absorbance = X ppm  
Percentage Zinc in the fertilizer =  $20(X)$

c) Precautions

- (1) Weighing must be done on an electric balance.
- (2) All the glass apparatus to be used should be of corning make and washed with dilute hydrochloric acid (1:4) and washed thoroughly with distilled and then with demineralised water.
- (3) The pipette should be rinsed with the same solution to be measured.
- (4) The outside of the pipette should be wiped with filter paper after taking out from the solution to be measured.
- (5) After using the pipette, place them on a clean dry filter paper in order to prevent contamination.
- (6) To start filtration, only a few drops should be added first in order to the one filter paper and then continue further filtration.

(iii) Determination of Magnesium

(a) Reagents

(1) Strontium chloride.- Dissolve 7.5 g of strontium chloride ( $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ ) in one litre of glass distilled water.

(2) Standard Magnesium solution.- Weigh 0.507g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and the funnel with glass distilled or demineralised water. This is 50 ppm Mg solution. Dilute 10 ml of 50 ppm solution of Mg to 100 ml to get 5 ppm standard Mg solution.

(3) Preparation of working standards.- Pipette the following volume of 5 ppm standard Mg solution in 50 ml numbered volumetric flasks. Add 10 ml of strontium chloride solution to each flask and make up the volume to 50 ml.

Flask No.	Volume of 5 ppm Mg solution taken (ml)	Volume of strontium chloride added (ml)	Concentration of magnesium after making the volume to 50 ml (ppm)
1	0.0	10.0	0.0
2	2.0	10.0	0.2
3	4.0	10.0	0.4
4	6.0	10.0	0.6

1	2	3	4
5	8.0	10.0	0.8
6	10.0	10.0	1.0

Stopper the flask and shake them well. Prepare fresh standards every fortnight.

(b) Procedure

(1) Pipette 20 ml of the solution which was prepared for the determination of zinc by dissolving 0.25 g of the fertilizer sample in one litre flask (Step A-2.2.1). Add 10 ml of strontium chloride. Make up the volume to 50 ml.

(2) Flame the standards and the samples on atomic absorption spectro-photometer at a wavelength of 285.5 nm (Mg line of the instrument).

Calculations

Prepare a standard curve of known concentrations of Mg solutions by plotting the absorbance value on Y-axis against their respective concentration values on X-axis. Percentage magnesium in the zinc fertilizer will correspond to the concentration values calculated from the standard curve.

Example :

Weight of the fertilizer ..... = 0.25 g  
Volume made ..... = 1000 ml  
Further dilution ..... = 2.5 times  
Reading of the sample from atomic absorption spectro-photometer = Y

Corresponding concentration of Mg from standard curve against Y absorbance ..... = X ppm  
Percentage magnesium in the fertilizer = X

(iv) Determination of copper.-

(a) Reagents

(1) Standard Copper Solution.- weigh 0.1965 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and the funnel with glass distilled water. Add one ml of 10 per cent sulphuric acid and make up the volume upto the mark. Stopper the flask and shake the solution well. This is 50 ppm Cu solution and should be stored in a clean bottle for further use. Dilute 10 ml of 50 ppm solution of copper to 100 ml to get 5 ppm standard copper solution.

(2) Glass distilled or mineralized acidified water of  $\text{pH} 2.5 \pm 0.5$  [same as given in 8(ii)(a)(2)]

(3) Preparation of working standards.- Pipette the following volume of 5 ppm standard copper solution in 50 ml numbered volumetric flasks and make the volume with acidified water.



Flask No.	Values of 5ppm standard Cu solution taken (ml)	Concentration of copper after making volume to 50ml (ppm)
1	0.0	0.0
2	2.0	0.2
3	4.0	0.4
4	6.0	0.6
5	8.0	0.8
6	10.0	1.0

Stopper the flasks and shake them well. Prepare fresh standards every fortnight.

(b) Procedure

(1) The solution which was prepared for the determination of zinc by dissolving 0.25 g of the fertilizer sample in one litre flask Step 8 (ii)(b)(1) should be used for the determination of copper.

(2) Flame the standards and the samples on an atomic absorption spectrophotometer at a wavelength of 324.8 mu (Cu line of the instrument).

Calculation

Prepare a standard curve of known concentrations of copper solutions by plotting the absorbance values on Y-axis against their respective concentration values on X-axis. Calculate the percentage copper in the zinc fertilizer by multiplying the copper concentration value calculated from the standard curve by 0.4.

Example:

Weight of the fertilizer samples. = 0.25g  
Volume made ..... = 1000ml

Reading of the sample from atomic absorption spectrophotometer ..... = Y

Corresponding concentration of copper from standard curve against Y absorbance .... = X ppm

Percentage copper in the fertiliser ..... = 0.4 X

(v) Determination of lead.-

(a) Reagents

- (1) Standard Lead Solutions.- Weigh 0.1599 g of lead nitrate  $[Pb(NO_3)_2]$  on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add 10ml of concentrated distilled nitric acid and make the volume upto the mark. Stopper the flask and shake the solution well. This is 100 ppm lead solution and should be stored in a clean bottle for further use. Dilute

10 ml of 100 ppm solution of lead to 100 ml with 1% nitric acid solution to get 10 ppm standard lead solution.

- (2) 1 per cent nitric acid solution.-Dilute 10 ml of concentrated distilled nitric acid to one litre with glass distilled water.

- (3) 20 per cent zinc sulphate solution.-Weigh 20 g of zinc sulphate  $(ZnSO_4 \cdot 7H_2O)$  and dilute to 100 ml with 1 per cent nitric acid solution.

- (4) Preparation of working standards.-Pipette the following volume of 10 ppm standard lead solution in 50 ml numbered volumetric flasks. Add 5 ml of 20 per cent zinc sulphate solution to each flask and make the volume with 1 per cent nitric acid solution.

Flask No.	Volume of 10 ppm lead solution taken (ml)	Volume of 20% zinc sulphate solution added (ml)	concentration of lead after making the volume to 50 ml (ppm)
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1	0.0	5.0	0.0
2	2.0	5.0	0.4
3	4.0	5.0	0.8
4	6.0	5.0	1.2
5	8.0	5.0	1.6
6	10.0	5.0	2.0

Stopper the flasks and shake them well.

(b) Procedure

- (1) Preparation of zinc sulphate fertiliser samples.-Weigh 1 g of the material on a clean watch glass and transfer to 50 ml volumetric flask through the funnel giving washings with 1 per cent nitric acid solution. Dissolve the material and make the volume with 1 per cent nitric acid solution. Samples should be prepared in duplicate.

- (2) Flaming the solutions.-Flame the standards and the samples on atomic absorption spectrophotometer at a wavelength of 217 mu (Lad line of instrument).

- (3) Calculations.-Prepare a standard curve of known concentrations of lead solution by plotting the absorbance values on Y-axis against their respective lead concentration on X-axis. Calculate the percentage lead in zinc fertilizer by multiplying lead concentration value calculated from standard curve by 0.005.

(vi) Determination of pH

(a) Procedure

Dissolve 5 gm of the material in

freshly boiled water. Dilute to 25 ml and mix. Determine the pH value of the solution with pH meter.

(vii) Determination of matter insoluble in water.-

(a) Procedure

Dissolve 25.0 g of the material in 125 ml of water. Filter through a weighed and prepared Gooch crucible or sintered glass crucible (G.No.4) and wash the residue thoroughly with water. Dry the crucible at  $110^{\circ} \pm 8^{\circ}$  to constant mass.

Calculations

Matter insoluble in water per cent by weight =  $4A$ . Where  $A$  = Weight in g of the residue.

NOTE.-In case a sample has been analysed by both the methods, viz, indicated under the heading '7' and '8' the result obtained by the method indicated under the heading '8' shall prevail.

9. Method of analysis of manganese sulphate, agricultural grade.

(i) Quality of Reagents.

Unless specified otherwise, pure chemicals and glass distilled or demineralized water shall be used in tests.

Note : 'Pure chemicals' means chemicals that do not contain impurities which affect the results of analysis. 'Demineralized water' means the water obtained after passing distilled water through a cation and an anion exchange resins or a combined cation anion exchange resin.

(ii) Determination of Manganese

(a) Reagents:

- (1) Standard Manganese Solution.- Weigh 3.0763 g of manganese sulphate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ -A.R. grade) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with acidified water and make the volume up to the mark. This solution will be 1000 ppm Mn. A secondary dilution of 5 ml to 100 ml with acidified water gives a 50 ppm working standard.
- (2) Glass distilled or demineralized water of pH  $2.5 \pm 0.2$ .-Dilute 1 ml of 10 per cent sulphuric acid to one litre with glass

distilled or demineralized water and adjust the pH to 2.5 with a pH meter using 10%  $\text{H}_2\text{SO}_4$  or NaOH. This solution is called acidified water.

- (3) Preparation of working standards.- Pipette the following volume of working standard solution in 50 ml numbered volumetric flasks and make the volume with acidified water.

Flask No.	Volume of Working standard taken (ml)	Concentration of manganese after making the volume to 50ml (ppm)
1	0.0	0.0
2	0.5	0.5
3	1.0	1.0
4	1.5	1.5
5	2.0	2.0
6	2.5	2.5
7	3.0	3.0
8	3.5	3.5
9	4.0	4.0

Stopper the flasks and shake them well. Prepare the standard in duplicate. The same acidified water should be used for preparing the solution of unknown fertiliser samples. Fresh standards should be prepared everytime when a fresh lot of acidified water is prepared.

(b) Procedure

- (1) Preparation of manganese sulphate fertiliser samples.-Weigh exactly 0.4 g of the material on the clean watch glass and transfer it to a one litre volumetric flask through the funnel giving repeated washings with acidified water and dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well.
- (2) Take 5 ml of the prepared solution in 250 ml volumetric flask and make the volume with acidified water. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flasks. The flasks should be rinsed with a 10 to 15 ml of the filtered and then continue filtration.

- (3) Flaming the solutions.-Flame the standards and the filtered samples on atomic absorption spectrophotometer at a wavelength of 279.5 mu (Mn. line of the instrument).

#### Calculation

Prepare a standard curve of known concentration of manganese solution by plotting the absorbance value on Y-axis against their respective manganese concentrations on X-axis. Calculate the percentage manganese in manganese fertiliser by multiplying manganese concentration value calculated from the standard curve by 12.5.

#### (c) Precaution

- (1) Weighing must be done accurately with a precision balance.
- (2) All the glass apparatus to be used should be corning/pyrex make and washed with dilute hydrochloric acid (1 :4) and washed thoroughly with distilled and then with demineralised water.
- (3) The pipette should be rinsed with the same solution to be measured.
- (4) The outside of the pipette should be wiped with filter paper after pipetting the solution to be measured.
- (5) After using the pipette, place them on a clear dry filter paper, in order to prevent contamination.
- (6) To start filtration, only a few drops should be added first in order to wet the filter paper and then continue further filtration.

#### 10. Alternative Method of Analysis of Manganese Sulphate Agricultural Grade.

##### (i) Quality of Reagents

Unless specified otherwise, pure chemicals and glass distilled or demineralized water shall be used in tests.

Note: 'Pure Chemicals' mean chemicals that do not contain impurities which affect the results of analysis.

"Demineralised water" means the water obtained after passing distilled water through a cation and anion exchange resins or a combined cation-anion exchange resin.

#### (ii) Determination of Manganese

##### (a) Reagents

- (1) Hydrogen peroxide ( $H_2O_2$ )-30 per cent.
- (2) Nitric acid ( $HNO_3$ )-concentrated
- (3) Orthophosphoric acid ( $H_3PO_4$  from steel industry)-85 percent
- (4) Potassium periodate ( $KIO_4$ ).
- (5) Glass distilled or mineralised water of pH  $2.5 \pm 0.5$ .

Dilute 1 ml of 10 per cent  $H_2SO_4$  to one litre with glass distilled or demineralised water and adjust the pH to 2.5 with a pH meter using 10%  $H_2SO_4$  or NaOH.

- (6) Standard manganese solution: Weigh 3.0763 g of manganese sulphate ( $MnSO_4 \cdot H_2O$ -AR. grade) on a clear watch glass and transfer it to one litre flask through the funnel giving several washings to the watch glass and the funnel with acidified water and make the volume up to the mark. This solution will be 1000 ppm  $Mn$ . A secondary dilution of 5 ml to 100 ml with acidified water gives a 50 ppm working standard.

- (7) Preparation of working standards: Pipette 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 ml of standard solution of manganese in a series of 11 different 100 ml beakers. Evaporate the solutions to dryness at low temperature on a hot plate or steam bath. Add 5 ml of  $HNO_3$  and 2 ml of 30%  $H_2O_2$ . Cover the beakers with watch glass and digest the contents on a steam bath or hot plate for 30 minutes. Remove the glass cover and evaporate the contents to dryness. Cool the beakers, add 2 ml of  $HNO_3$ , 5 ml of phosphoric acid and heat till boiling. Again cool and mix the contents with 10 ml of demineralised

water. Add 0.3 of KIO<sub>4</sub> and heat the solutions on water bath until pink colour appears. Add about 20 ml water and heat for about 40 minutes or until there is no more increase in colour intensity. Cool the beakers to room temperature, transfer the solutions to 50 ml volumetric flasks. Make the volume with deionised water and shake thoroughly. The concentration of manganese ion will be as follows.

Flask No.	Volume of working standard taken(ml)	Concentration of manganese after making the volume to 50ml(ppm)
1.	0.0	0.0
2.	0.5	0.5
3.	1.0	1.0
4.	1.5	1.5
5.	2.0	2.0
6.	2.5	2.5
7.	3.0	3.0
8.	3.5	3.5
9.	4.0	4.0
10.	4.5	4.5
11.	5.0	5.0

Measure the light transmittance at 540 mu (green filter). Make the standard curve by plotting per cent transmittance against the concentration of manganese.

(b) Procedure

(1) Analysis of manganese sulphate fertiliser-sample.-Weigh exactly 0.500g of the material on a clean watch glass and transfer it to a one litre volumetric flask through the funnel giving repeated washings with acidified water and dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well. Pipette one ml solution in a 100 ml pyrex/earning breaker and proceed further as described above for preparation of standard curve and make the volume to 50 ml. The standard and the test samples should be prepared and processed for estimation at the same time.

Calculation

The percentage of manganese in the manganese sulphate fertiliser sample is calculated by multiplying manganese concentration value calculated from standard curve by 10.

EMBLEM  
FORM A

(See Clause 8)

FORM OF APPLICATION TO OBTAIN  
DEALER'S (WHOLESALE OR RETAIL)  
CERTIFICATE OF REGISTRATION.

To

The Registering Authority  
Place  
State of

1. Full name and address of the applicant:  
(a) Name of the concern, and postal address:  
(b) Place of business (Please give exact address):  
(i) for sale  
(ii) for storage
2. Is it a proprietary/partnership/limited company/Hindu Undivided Family concern? Give the name(s) and address(es) of proprietor/partners/manager/Karta:-
3. In what capacity is this application filed.  
(i) Proprietor,  
(ii) Partner,  
(iii) Manager,  
(iv) Karta.
4. Whether the application is for wholesale or retail dealership?
5. Have you ever had a fertiliser dealership registration certificate in the past? If so give the following details :-  
(i) Registration number-  
(ii) Place for which granted-  
(iii) Whether wholesale or retail dealership-  
(iv) Date of grant of registration certificate-  
(v) Whether the registration certificate is still valid?-  
(vi) If not, when expired?-

- (vii) Reasons for non-renewal-
- (viii) If suspended/cancelled and if so, when-
- (ix) Quantity of fertilisers, handled during last year-
- (x) Names of products handled--
- (xi) Name of source of supply of fertilisers.-

6. Was the applicant ever convicted under the Essential Commodities Act, 1955, or any Order issued thereunder, including the Fertiliser (Control) Order, 1957, during the last three years preceding the date of application? If so give details.

7. Give the details of the fertilisers to be handled:

S.No.	Name of Fertiliser	Source of supply
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8. Please attach certificate(s) of source from the supplier(s) indicated under column 3 of Sl.No. 7.

9. I have deposited the registration fee of Rs..... vide Challan No....., dated..... in treasury/Bank.

10. Declaration:-

(a) I/We declare that the information given above is true to the best of my/our knowledge and belief, and no part thereof is false.

(b) I/We have carefully read the terms and conditions of the Certificate of Registration given in form 'B' appended to the Fertiliser (Control) Order, 1985, and agree to abide by them.

Dated: \_\_\_\_\_ Signature of the Applicant(s)

Place: \_\_\_\_\_

Note:-1. Where the business of selling fertilisers is intended to be carried on at more than one place, a separate application should be made for registration in respect of each such place.

2. Where a person intends to carry on the business of selling fertilisers both in retail and wholesale, separate applications for retail and wholesale business should be made.

3. Where a person represents or intends to represent more than one State Government,

Commodity Board, Manufacturer or Wholesale dealer, separate certificate of source from each such source should be enclosed.

For use in Office of Registering Authority.

Date of receipt \_\_\_\_\_ Name and designation of Officer receiving the application.

# EMBLEM

## FORM 'B'

(See Clause 9)

Book No..... Registration No.....  
Date of Issue .....  
Valid upto .....

## CERTIFICATE OF REGISTRATION TO CARRY ON THE BUSINESS OF SELLING FERTILISERS AS A WHOLESALE/RETAIL DEALER IN THE STATE OF .....

..... is hereby granted certificate of registration to carry on the business of selling fertilisers in retail/wholesale at the place specified below in the State of ..... subject to the terms and conditions specified below and to the provisions of the Fertiliser (Control) Order, 1985.

## DESCRIPTION OF THE PLACE AND TYPE OF BUSINESS

Name and style by which the business is carried on. \_\_\_\_\_ Location of sale depot.

Location of godowns Type of fer- Source of supply attached to sale depot. tiliser.

Date: \_\_\_\_\_ Registering Authority Seal: \_\_\_\_\_ State of.....

Terms and conditions of certificate of registration:

1. This certificate of registration shall be displayed in a prominent and conspicuous place in part of the business premises open to the public.

2. The holder of the certificate shall comply with the provisions of the Fertilisers (Control) Order, 1985 and the notification issued thereunder for the time being in force.

3. The certificate of registration shall come into force immediately and be valid upto .....  
UNLESS PREVIOUSLY CANCELLED OR SUSPENDED.

4. The holder of the certificate shall from time to time report to the registering Authority any change in the premises of sale depot and godowns attached to sale depot.

5. The dealer shall submit a report to the Registering Authority, with a copy to the Block Development Officer or such other officer as the State Government may notify, in whose jurisdiction the place of business is situated, by the 5th of every month, showing the opening stock, receipts, sales and closing stocks of fertilisers in the preceding month. He shall also submit in time such other returns as may be prescribed by the Registering Authority.

Note: 1. The original is meant for the holder of the certificate which will be delivered against his proper and adequate acknowledgement. The original certificate of registration shall be torn off at the place performed while all duplicate shall be kept intact bound in the registration book by the Registering Authority.

2. Where the business of selling fertilisers is intended to be carried on at more than one place, a separate registration certificate should be obtained in respect of each such place.

3. Where a person intends to carry on the business of selling fertilisers both in retail and wholesale, separate registration certificate should be obtained for retail and wholesale business.

# EMBLEM

## FORM 'C'

(See Clause 11)

### APPLICATION FOR RENEWAL OF THE CERTIFICATE OF REGISTRATION TO CARRY ON THE BUSINESS OF SELLING FERTILISERS IN RETAIL/WHOLESALE.

To

The Registering Authority  
Place  
State

I/We hereby apply for renewal of the certificate of registration to carry on the business of selling fertilisers in retail/wholesale under the name and style of .....  
The certificate of registration desired

to be renewed was granted by Registering Authority for the ..... (mention place and state) and allotted Registration Certificate No..... on the ..... day of ..... 19 .....

2. (1) I/We hereby declare that the situation on my/our premises where fertilisers are (a) stored and (b) sold is as stated below:-

(a)	(b)
Premises where fertilisers are stored.	Premises where fertiliser are sold.

(ii) I/We hereby declare that the fertiliser(s) in which I/We am/are carrying on the business of selling and the name(s) of manufacturer(s), Commodity Board(s), State Government(s) and whole-sale dealer whom I/We represent are as stated below:

(a)	(b)
Name(s) of fertiliser(s)	Name(s) of manufacturer(s), Commodity Board(s), whole sale-dealer(s)

3. I/We enclose a certificate of source from the manufacturer(s), commodity board(s), State Government(s), wholesale dealer(s) whom I/We represent or intend to represent and from whom fertiliser(s) will be obtained by me/us.

Full name and address of the applicant(s) (in block letters)	Signature of applicant(s)
--	---------------------------

Date :

Place :

Certified that the certificate of registration bearing number .... granted on for the period from ..... to carry on the business of selling fertilisers in retail/wholesale at the premises situated at ..... is hereby renewed till the ....., unless previously suspended or cancelled under the provisions of the Fertiliser (Control) Order, 1985.

(a)	(b)
Location of sale depot	Location of godowns attached to sale depot, where fertiliser will be stored.

(c)	(d)
Name(s) of fertiliser(s) for which certificate of registration is renewed.	Name(s) manufacturer(s) Commodity Board(s) State Government (s), wholesale dealer(s) whom the dealer represents.
Date :	Registering Authority
Renewal number:	Place:
Seal:	State:

**EMBLEM  
FORM 'D'**

[See Clause 14(2) (a) & 18(1)]

**FORM OF APPLICATION TO OBTAIN A CERTIFICATE OF MANUFACTURE OF PHYSICAL/GRANULATED MIXTURE OF FERTILISERS/RENEWAL.**

To

The Registering Authority,

Place .....

State of .....

1. Full name and address of the applicant:
2. Does the applicant possess the qualification prescribed by the State Government under sub-clause (1) of clause 14 of the Fertiliser (Control) Order, 1985:
3. Is the applicant a new comer ? (Say 'Yes' or 'No'):
4. Situation of the applicant's premises where physical/granulated mixture will be prepared.
5. Full particulars regarding chemical analysis of the physical/granulated mixture of fertilisers for which the certificate is required and the raw materials used in making the mixture:
6. Full particulars of any other certificate of manufacture, if any, issued by any other Registering Authority:
7. How long has the applicant been carrying on the business of preparing physical granulated mixture of fertilisers:
8. Quantities of each physical/granulated mixture of fertilisers (in tonnes) in my/our possession on the date of the application and held at different addresses noted against each:

9. (i) If the applicant has been carrying on the business of preparing physical/granulated mixtures of fertilisers give all particulars of such mixtures handled, the period and the place(s) at which the mixture of fertilisers was done:

(ii) Also give the quantities of physical/granulated fertiliser mixtures handled during the past calendar year:

10. If the application is for renewal indicate briefly why the original certificate could not be acted on within the period of its validity.

11. I have deposited the prescribed registration certificate fee/renewal fee:

Declaration:

(a) I/We declare that the information given above is true and correct to the best of my/our knowledge and belief, and no part thereof is false.

(b) I/We have carefully read the terms and conditions of the certificate of manufacture given in Form F appended to the Fertiliser (Control) Order, 1985 and agree to abide by them.

(c) I/We declare that the physical granulated mixture for which certificate of manufacture is applied for shall be prepared by me/us or by a person having such qualifications as may be prescribed by the State Government from time to time or by any other person under my/our direction, supervision and control or under the direction, supervision and control of person having the said qualifications.

Signature of the applicant(s)

Name and address of applicant in block letters:

Date:

Place :

**EMBLEM**

**FORM 'E'**

[See Clause 14(2)(b)]

**FORM OF APPLICATION TO OBTAIN A  
CERTIFICATE OF MANUFACTURE FOR  
SPECIAL MIXTURE OF FERTILISERS**

To

The Registering Authority  
Place  
State

Name and address of the  
applicant(s) in block letters:  
  
Date :  
Place:

**EMBLEM**

**FORM 'F'**

[See Clause 15(2) & 18(2)]

1. Full Name and address of the applicant:
2. Does the applicant possess qualifications prescribed by the State Government under sub-clause (1) of 14 of the Fertiliser (Control) Order, 1985:
3. Name and address of the person requiring the special mixture of fertilisers:
4. Particulars of certificate(s) of manufacture already obtained from the same Registering Authority:
5. Situation of the applicant's premises where fertilisers are/will be mixed:
6. Full particulars regarding chemical analysis of the special mixture of fertilisers required to be manufactured and the materials used in making the special mixture.
7. I am enclosing an attested copy of the requisition made by the purchaser of the special mixture of fertilisers:
8. I have deposited the prescribed registration certificate fee:

Book No.....Certificate No.....  
Date of issue.....  
Valid upto.....

**CERTIFICATE OF MANUFACTURE IN  
RESPECT OF PHYSICAL/GRANULATED  
MIXTURE OF FERTILISER**

..... is hereby given the certificate for manufacture of the physical/granulated mixture of fertiliser specified below subject to the terms and conditions of this certificate and to the provisions of the Fertiliser (Control) Order, 1985.

Full particulars of the mixture of fertilisers	Full address of the premises where the mixture of fertiliser will be made.
	Registering Authority State

**Declaration :**

- (a) I/We declare that the information given above is true and correct to the best of my/our knowledge and belief, and no part thereof is false.
- (b) I/We have carefully read the terms and conditions of the certificate of manufacture given in form 'G' appended to the Fertiliser (Control) Order, 1985 and agree to abide by them.
- (c) I/We declare that the special mixture for which a certificate of manufacture is applied for, shall be prepared by me/us or by a person having such qualification as may be prescribed by the State Government from time to time or by any other person under my/our direction, supervision and control or under the direction, supervision and control of a person having the said qualifications.

Date :  
Seal :

Renewed upto.....

Registering Authority.  
State.

Date :  
Seal :

**Terms and conditions of this certificate:**

1. The holder of this certificate shall display the original thereof in a conspicuous place open to the public in part of the principal's premises in which the business of making the physical/granulated mixture of fertilisers is carried on and also a copy of such certificate in similar manner in every other premises in which that business is carried on. The required number of copies of the certificate shall be obtained on pay-

Signature of the applicant(s)



ment of the fees thereof.

2. The holder of this certificate shall not keep in the premises in which he carries on the business of making physical/granulated mixture of fertilisers, any mixture of fertilisers, in respect of which a certificate of registration has not been obtained under the Fertiliser (Control) Order, 1985.
3. The holder of this certificate shall comply with the provisions of the Fertiliser (Control) Order, 1985 and the notification order and directions, issued thereunder for the time being in force.
4. The holder of the certificate shall report forthwith to the Registering Authority any change in the premises specified in the certificate or any new premises in which he carried on the business of making physical/granulated mixture and shall produce before the authority the original certificate and copies thereof so that necessary corrections may be made therein by that authority.
5. The holder of this certificate shall ensure that the physical/granulated mixture in respect of which a certificate of registration has been obtained is prepared by him or by a person having such qualifications, as may be prescribed by the State Government from time to time or by any other person under the directions, supervision and control of the holder or the person having the said qualifications.
6. The certificate and copies thereof, if any, will be machine numbered and delivered against the signature of the holder thereof or his agent on the carbon copy of the certificate which will be kept intact bound in the "Certificate Book" by each Registering Authority.

**EMBLEM**

**FORM 'G'**

[See Clause 15(2)]

Book No. .... Certificate No....  
Date of issue....

**Certificate of manufacture  
in respect of the  
Special mixture of fertilizers**

..... is/are hereby given this certificate for the preparation of..... tonnes of special mixture of fertiliser specified below subject to the terms and conditions of this certificate, and to the provisions of the Fertiliser (Control) Order, 1985.

Full Particulars of the special mixture of fertilisers

2. The certificate is valid up to .....

Full address of the premises where the special mixture of fertilisers will be made.

Registering Authority:

State :

Date :

Seal :

Terms and conditions of this certificate:

1. The holder of the certificate shall display the original thereof in a conspicuous place open to the public in a part of the principal premises in which the business of making the special mixture of fertilisers is carried on and also a copy of such certificate in similar manner, in every other premises in which that business is carried on. The required number of copies of the certificate shall be obtained on payment of fees therefor.
2. The holder of this certificate shall not keep in the premises in which he carries on the business of making special mixture of fertilisers, any mixture of fertilisers in respect of which a certificate of manufacture has not been obtained under the Fertiliser (Control) Order, 1985.
3. The holder of the certificate shall comply with the provisions of the Fertiliser (Control) Order, 1985 and the notification, orders and directions issued thereunder for the time being in force.
4. The holder of this certificate shall report forthwith to the Registering Authority any change in the premises specified in the certificate or any new premises in which he carries

on the business of making the special mixture and shall produce before the authority the original certificate and copies thereof so that necessary corrections may be made therein by that authority.

5. The holder of this certificate shall ensure that the special mixture in respect of which a certificate of manufacture has been obtained is prepared by him or by a person having such qualifications as may be prescribed by the State Government from time to time or any other person under the direction, supervision and control of the holder or person having the said qualifications.

Note:- The certificate and copies thereof, if any, will be machine numbered and delivered against the signature of the holder thereof or his agent on the carbon copy of the certificate which will be kept intact bound in the "Certificate Book" by each Registering Authority.

**EMBLEM**

**FORM 'H'**

[See Clause 23(1)(b)]

**APPLICATION FOR GRANT OF PERMISSION  
TO SELL NON-STANDARD  
FERTILISERS**

To  
The Registering Authority,  
State of .....

1. Full name and address of the applicant:
2. The name of fertilisers in which the applicant has been authorised to carry on business:
3. Number and date of registration certificate granted in favour of the applicant:
4. The particulars of non-standard fertilisers which are intended to be sold:

(i) Name of the fertiliser:

(ii) The source from which the fertilisers were originally purchased and the period during which they have been held by the applicant:

(iii) Brief reasons leading to the material having become non-standard:

(iv) The chemical specifications as analysed by an authorised chemical laboratory:

(v) Name and appearance of the non-standard fertilisers:

Note:- Chemical analysis to be indicated shall be one which has been certified by an authorised chemical laboratory on the basis of the samples drawn, sealed and despatched in the presence of the local Inspector of Fertilisers or anybody authorised to inspect the fertilisers.

I enclose a copy of the certificate of registration for dealing in fertilisers already granted to me and I have deposited the prescribed fee for permission to sell non-standard fertilisers.

I certify that I will comply with the requirements of sub-clause(a) of clause 23 of the Fertiliser (Control) Order, 1985 which requires that each container of non-standard fertilisers is to be conspicuously superscribed with the words 'Non-standard' and also with the sign 'X', both in red colour.

Signature of the applicant.

Name and address of applicant  
in block letters

Date :

Place :

**EMBLEM**

**FORM 'I'**

[See Clause 23(1)(b)]

Book No. ....

Date of issue .....

Valid upto .....

Authorisation for the sale of non-standard fertilisers:

M/s ..... is/are hereby permitted to sell the non-standard fertilisers described below subject to the terms and conditions noted hereunder:

Particulars of non-standard fertilisers:-

1. Name of the fertiliser.
2. Detailed chemical specifications including the nutrient content.
3. Nature and appearance of the non-standard fertiliser.

4. Place of location of non-standard fertiliser.

9. Name & Address of Fertiliser Inspector drawing sample \_\_\_\_\_

Terms and conditions:

1. The seller of the non-standard fertiliser described above shall comply with the provisions of sub-clause (a) of Clause 23 of the Fertiliser(Control) Order, 1985.
2. The non-standard fertilisers described above shall be sold at a price not exceeding ..... per metric ton or ..... per bag of ..... kgs.
3. The non-standard fertiliser described above shall be sold before the expiry of the validity period mentioned above or before the expiry of the extended period, if any.

Signature & Metallic Seal  
impression of Fert. Inspector

**RECEIPT OF THE DEALER**

Certified that the sample of fertiliser has been drawn in accordance with the procedure laid down in the Fertiliser(Control) Order, 1985 from the stock in my possession, and I have signed the test samples at the time of wax sealing. I have also received one test sample out of the three test samples prepared.

Signature of dealer/  
manufacturer/Pool handling  
agency with address

Registrations Authority  
State of .....

Date :

Seal :

Signature & Seal of  
Fertiliser Inspector

**EMBLEM**

**FORM 'J'**

[See Clause 28(1)(b)]

**FORM INDICATING PARTICULARS OF  
FERTILISER SAMPLED**

1. Name & Address of dealer/manufacturer- \_\_\_\_\_
2. Date of sampling \_\_\_\_\_
3. Details of markings on bags from where sample has been taken \_\_\_\_\_
  - (i) Type & grade of fertiliser with brand \_\_\_\_\_
  - (ii) Name of manufacturers \_\_\_\_\_
  - (iii) Registration No. \_\_\_\_\_
  - (iv) Lot No. & date of manufacture \_\_\_\_\_
  - (v) Composition \_\_\_\_\_
4. Date of receipt of the stock by the dealer/manufacturer/Pool handling agency \_\_\_\_\_
5. Serial No./Code No. of sample \_\_\_\_\_
6. Stock position of the lot \_\_\_\_\_
7. Physical condition of fertiliser \_\_\_\_\_
8. Whether samples drawn from open bags or stitched bags \_\_\_\_\_

**EMBLEM**

**FORM 'K'**

[See Clause 30(1)]

**MEMORANDUM TO ACCOMPANY FERTI-  
LISER SAMPLE FOR ANALYSIS**

No.

From

To

Incharge

Fertiliser Quality Control Laboratory

The fertiliser samples as per details given below are sent for analysis:-

- (1) Name of fertiliser, grade and brand .....
- (2) Date of sampling .....
- (3) No. of samples taken .....
- (4) Serial No. of sample .....
- (5) Physical condition of fertiliser.
- (6) Code Number of Sample .....

2. The analysis report may please be forwarded to .....

Place:

Date: Signature of Fertiliser Inspector

EMBLEM

FORM 'L'

[See Clause 30(2)]

ANALYSIS REPORT OF FERTILISER

SAMPLE

No.

Government of

(Name of the Laboratory)

Date :

To

The Fertiliser Inspector

The analysis report of the fertiliser sample forwarded vide your reference No. .... dated ..... is as per details given below:-

1. Name of fertiliser, grade and brand .....
2. Date of sampling .....
3. Serial No. of sample as indicated by the Inspector .....
4. Code No. of sample as indicated by the Inspector .....
5. Date of receipt of the sample in the laboratory .....
6. Laboratory sample No. ....
7. Date of analysis of sample .....
8. Chemical analysis of fertiliser (on fresh weight basis except in the case of Urea on dry weight basis) .....

S. No.	Specification as per F.C.O.	Composition as per analysis	Variation	Permissible tolerance limit
--------	-----------------------------	-----------------------------	-----------	-----------------------------

1 2 3 4 5

- (i) Moisture
- (ii) Total N
- (iii)  $\text{NH}_4\text{N}$
- (iv)  $\text{NH}_4 + \text{NO}_3\text{N}$
- (v) Urea N
- (vi) Total  $\text{P}_2\text{O}_5$
- (vii) Nitrate Ammonium
- (viii) Citrate soluble  $\text{P}_2\text{O}_5$
- (viii) Citric Acid soluble  $\text{P}_2\text{O}_5$
- (ix) Water soluble  $\text{P}_2\text{O}_5$
- (x) Water soluble  $\text{K}_2\text{O}$
- (xi) Particle size
- (xii) Others

Remarks:- The sample is/is not according to specifications.

Signature of the Incharge  
Fertiliser Quality Control  
Laboratory

Copy to :-

Director of Agriculture,

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[No. 11-3/83-STU]

J.K. ARORA, jt. Secy.

DEPARTMENT OF AGRICULTURE

Order

10/1/79-AGR-VII

The following orders by the Government of India Ministry of Agriculture, (Deptt., of Agri & Coop.) New Delhi, which were published in the Gazette of India (Extraordinary) Part-II, Section 3 Sub-Section(ii) are hereby republished in the Official Gazette for general information of the public.

- (i) Order No.724(E) dated 28/7/88  
(ii) Order No.725(E) dated 28/7/88  
( A. P. Panvelkar )  
Under Secretary (Agri.)

No.1-4/88-Fert.Law  
Government of India  
Ministry of Agriculture  
Department of Agriculture & Coopn.

New Delhi, the 28th July, 1988.

ORDER

S.O. 724(E). In exercise of the powers conferred by section 3 of the Essential Commodities Act, 1955, (10 of 1955), the Central Government hereby, makes the following order further to amend the Fertiliser (Control) Order, 1985, namely:-

1. (1) This order may be called Fertiliser(Control) (Second Amendment) Order, 1988.  
(2) It shall come into force on the date of its publication in the Official Gazette.
2. In Schedule I to the Fertiliser (Control) Order, 1985, under the heading "Part-A. Specifications of fertilisers" and sub-heading "1(d) N.P.Fertilisers" after serial number 11 relating to "Mono ammonium phosphate(11-52-0)" and the entries thereto, the following serial number and entries shall be inserted, namely:-

Name of Fertiliser	Specifications
1.	2.

"12. Nitrophosphate.  
23:23:0

- (i) Moisture percent by 1.5 weight,maximum.
- (ii) Total Nitrogen 23.0 percent by weight, minimum.
- (iii) Nitrogen in 11.5 ammoniacal form,percent by weight,minimum.
- (iv) Nitrogen in 11.5 nitrate form percent by weight, maximum.
- (v) Neutral ammonium 23.0 citrate soluble phosphate as(P<sub>2</sub>O<sub>5</sub>) percent by weight, minimum.
- (vi) Water soluble 18.5 phosphate as P<sub>2</sub>O<sub>5</sub> percent by weight, minimum.
- (vii) Calcium nitrate 1.0 percent by weight, maximum.
- (viii) Particle size: 90% of the material shall pass through 4 mm IS Sieve and be retained on 1 mm IS Sieve. Not more than 5 percent shall be below 1mm IS Sieve."

Sd/-  
(C. Ranga Rao)  
Joint Secretary to the Govt.of India.

NOTE:- The Fertiliser(Control) Order, 1985 was published vide G.S.R. 758(E) dated 25th September, 1985.

- (2) Schedule I,Part A of the Fertiliser(Control)Order, 1985, was amended vide:-
  - (i) G.S.R. 201 (E) dated 14th February, 1986.
  - (ii) G.S.R. 1160 (E) dated 21st October, 1986.
  - (iii) S.O. 822 (E) dated 14th September, 1987.
  - (iv) S.O. 1079 (E) dated 11th December, 1987.
  - (v) S.O. 252 (E) dated 11th March, 1988.

No.1-12/87-Fert.Law  
Government of India  
Ministry of Agriculture  
Department of Agriculture and  
Co-operation

...

New Delhi, the 28th July, 1988.

# O R D E R

S.O. 725 (E). In exercise of the powers conferred by section 3 of the Essential Commodities Act, 1955 (10 of 1955), the Central Government hereby makes the following order further to amend the Fertiliser(Control) Order, 1985, namely:-

1. (1) This order may be called the Fertiliser (Control) (Third Amendment) Order, 1988.  
(2) It shall come into force on the date of its publication in the Official Gazette.
2. In the Fertiliser(Control) Order, 1985 (hereinafter referred to as the said order), in clause 2,-
  - (i) in under clause (d), the words "essential plant shall be omitted;
  - (ii) sub-clause (g) shall be omitted;
  - (iii) for sub-clause(h), the following sub-clause shall be substituted, namely:-

'(h) "fertiliser" means any substance used or intended to be used as a fertiliser of the soil and/or crop and specified in Part-A of Schedule-I and includes a mixture of fertilisers, mixtures of micro-nutrient fertilisers and special mixture of fertilisers';
  - (iv) for sub-clause (j), the following sub-clause shall be substituted, namely:-

'(j) "grade" means the nutrient contents in the fertiliser expressed in percentage';
  - (v) in sub-clause (k) -
    - (a) the words "containing any two or more essential plant nutrients" shall be omitted;
    - (b) for the words "fertiliser materials", the word "fertilisers" shall be substituted;
  - (vi) in sub-clause (m), after the words "mixtures of fertilisers",

the words "or mixtures of micro-nutrient fertilisers" shall be inserted;

- (vii) after sub-clause (n) the following sub-clause shall be inserted, namely:-

'(na) "mixture of micro-nutrient fertilisers" means a mixture of two or more micro nutrient fertilisers, specified under sub-heading 1(f) of part-A of Schedule I.';

- (viii) in sub-clause (p), for the words "containing any two or more essential plant nutrient made by physically mixing", the words "made by physically mixing two or more" shall be substituted;

- (ix) in sub-clause (q), after the entry (ii), the following entry shall be inserted, namely:-

"(iii) in relation to a mixture of micro-nutrient fertilisers, the standard set out in respect of that mixture under sub-clause (2) of clause 13 by the State Government, subject to limits of permissible variation as specified in part B of schedule I";

3. In clause 12 of the said order:-

- (i) after the words "any mixture of fertilisers", the words "or mixtures of micro-nutrient fertilisers" shall be inserted;
- (ii) for the letters "16", the letters and word "15 or 16" shall be substituted;

4. For the clause 13 of the said order, the following clause shall be substituted, namely:-

## "13. Standards of mixtures of Fertilisers -

- (1) Subject to the other provisions of this order, no person shall manufacture any mixture of fertilisers unless such mixture conforms to the standards set out in the notification to be issued by the Central Government in the Official Gazette.
- (2) Subject to the other provisions of this order, no person shall manufacture any mixture of micro-nutrient fertilisers unless such mixture conforms to the standards set out in the notification to be issued by the State Government in the Official Gazette";

- (3) Notwithstanding any thing contained in this Order, those units which are already manufacturing mixtures of micro-nutrient fertilisers, as on the date of publication of this order, for which standards have not yet been specified under clause 13(2), may continue to manufacture for sale, sell, offer for sale, stock or exhibit for sale or distribute such mixtures of micro-nutrient fertilisers for a period of one year from such date or till the standards are prescribed by the State Governments whichever is earlier subject to the condition that before the expiry of said period, they will follow the standards prescribed by State Government and obtain Certificate of manufacture under clause 15".
- (4) No Certificate of Manufacture shall be granted in respect of any mixture of fertilisers or mixture of micro-nutrient fertilisers, which does not conform to the Standards set out in the notification referred in sub clause (1) or (2);
- (5) Nothing in this Clause shall apply to special mixtures of fertilisers".
5. In clause 14 of the said order, - in sub clause (1), sub clause (2) and item (a) of sub-clause (2) after the words "mixture of fertilisers", the words "or mixtures of micro-nutrients fertilisers", shall be inserted;
6. In Clause 15 of the said order, - (i) in the title, sub clause (1) and sub clause (2), after the words "mixture of fertilisers", the words "or mixture of micro nutrient fertilisers" shall be inserted;
- (ii) in sub-clause (1), for the word 'of' after the words "mixture of fertilisers", the word "or" shall be substituted;
7. In clause 17 of the said order, - in the title and the entries there under after the words "mixture of fertilisers", the words "or mixtures of micro nutrient fertilisers" shall be inserted;
8. In clause 18 of the said order, - in the title and in sub clause (1), after the words "mixtures of fertiliser", the word "or mixture of micro-nutrient fertilisers" shall be inserted;
9. In clause 19 of the said order, in sub-clause (1) (b),
- (i) after the words "mixture of fertilisers", the words "or mixture of micro-nutrient fertilisers" shall be inserted;
- (ii) after the words "prescribed standard", the words "(subject to such limits of permissible variation as may be specified from time to time by the Central Government)" shall be inserted;
- (iii) the words "(subject to such limits of permissible variation in relation to total nitrogen, neutral and Ammonium Citrate soluble phosphoric Acid ( $P_2O_5$ ), water soluble phosphoric Acid ( $P_2O_5$ ), water soluble potash ( $K_2O$ ), as may be specified from time to time by the Central Government" shall be omitted.
10. In clause 38 of the said order, after sub-clause (4), the following sub-clause shall be inserted, namely: - "(5) The State Government may by notification in the Official Gazette and on such terms and conditions as may be specified in such notification, constitute a Committee called the State Fertiliser Committee consisting of a chairman and not more than 5 other members, having experience or knowledge in the field, including a representative from State Agricultural University, the Fertiliser Industry and Indian Micro-Fertilisers Manufacturers Association to advise the State Government regarding the grades/formulations of mixture of micro-nutrient fertilisers and their specifications
11. In form D, to the said order wherever the words "mixture of fertilisers" occur, after them, the words "mixtures of micro-nutrient fertilisers" shall be inserted, -
12. In the form F, to the said order wherever the words "mixture of fertilisers occur, after them, the words "mixture of micro-nutrient fertilisers" shall be inserted.

Sd/-

( G. Ranga Rao )

Joint Secretary to the Government of India.